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TECHNICAL REPORT ARLCD-TR-80033

CURING AND MOLDING OF PLASTICS USED IN SCATTERABLE MINES

DONALD VANDERBILT

MAY 1981



US ARMY ARMAMENT RESEARCH AND DEVELOPMENT COMMAND
LARGE CALIBER
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20. ABSTRACT (Continue on reverse side if necessary and identify by block number) The effect of ATC-3 accelerator on the curing time of the epoxy encapsulating the ADAM scatterable mine was investigated by Honeywell, Inc. Curing time was reduced but so were physical properties, which became unacceptably low. Anhydrides, which did not have time to react in the presence of the ATC-3 accelerator and which acted as a plasticizer, are the presumed cause of the low values.		

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The curing effect of ultraviolet radiation on the polyurethane encapsulant for electronics in the GATOR, GEMSS, and RAAM scatterable mines was investigated in-house. Cure time was reduced, but this reduction resulted from heat effect alone. Cure temperatures were far above those allowable, and no photo initiation took place.

Mechanical and in situ bonding of the GATOR mine and its styrene aeroballistic section was studied in-house. Such bonding was favorably recommended as a cost-saving production measure, but it will not be used at existing mine production facilities because too much tooling used in common for the GATOR, GEMSS, and RAAM mines would have to be modified.

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INTRODUCTION

The scatterable mines (ADAM, GATOR, GEMSS, and RAAM) include relatively large amounts of plastics. The ADAM mine has an epoxy housing for the kill mechanism that also serves as an encapsulant for the mine electronics. The GATOR, GEMSS, and RAAM mines have polyurethane encapsulants for their electronics; the GATOR mine, in addition, has a styrene aeroballistic section. Because the encapsulants of all mines enclose heat-sensitive electronic components, they should not be heated externally during the cure periods to a temperature greater than 71.1°C (160°F). Because of this temperature limitation, these mines require extended cure times.

This investigation consisted of three separate phases. In phase 1, Honeywell, Inc. (contract DAAK-10-79-C-0398) determined the effect of ATC-3 accelerator in reducing the cure time of the epoxy used to form the housing of the ADAM mine. The second and third phases were done in-house. In phase 2 the effect of ultraviolet radiation on the cure time of the polyurethane encapsulant in the GATOR, GEMSS, and RAAM mines was investigated. In phase 3 new bonding methods for the GATOR mine and its styrene aeroballistic section were investigated with the object of simplifying the assembly procedure.

BACKGROUND

Scatterable ADAM antipersonnel mines are loaded in 155-mm M692 and M731 projectiles, which are fired from M109A1 and M198 howitzers. The mines are ejected from the projectiles in flight and fall freely to the ground. The mine housings, which are made of epoxy, must withstand the impacts of gun firing, ejection from the projectiles, and hitting the ground while protecting the mines electronics and kill mechanisms against damage.

Scatterable GATOR antipersonnel and antiarmor mines are delivered from canisters carried by aircraft. The mines fall freely, but their flight is controlled to a degree by styrene aeroballistic sections. Their electronics are encapsulated in polyurethane, which must protect them against rough handling and ground impacts.

Scatterable GEMSS antipersonnel and antiarmor mines are deployed by an M128 mine dispenser from a ground vehicle. Their electronics are protected in the same way as are those in the GATOR mines.

Scatterable RAAM antiarmor mines are loaded in 155-mm M718 and M741 projectiles, which are fired from M109A1 and M198 howitzers similarly to the ADAM mines. They are also ejected from the projectiles in flight and fall freely to the ground. Their electronics are protected by the polyurethane encapsulant against the impacts of gun firing, ejection from the projectiles, and hitting the ground.

PHASE 1 -- USE OF ATC-3 ACCELERATOR¹ IN CURING OF EPOXY FOR ADAM MINE HOUSING

Because the epoxy housing of the ADAM mine encapsulates heat-sensitive electronics, it should not be heated externally above 77.1°C (160°F); therefore the housing requires an extended cure time after it has been molded. Forty hours are required for the housing itself to cure, and an additional forty hours are required to seal the closing plug in the housing after the kill mechanism has been inserted. Honeywell, Inc. as contractor, investigated the effect of the ATC-3 accelerator on the cure time and the properties of the epoxy. It was considered possible that ATC-3 accelerator would not only speed up epoxy curing but that it might also completely replace the uranyl acetylacetone accelerator then in use. At \$5.00 per pound² for ATC-3 accelerator versus \$200 per pound for uranyl acetylacetone, a substantial cost saving might have been realized.

Procedure

Ingredients shown in table 1 were furnished by Hysol³, for this investigation. Epoxy control samples were made up in the ratio of two parts by weight of part A to one part of part B (table 2), heated to 132.2°C (150°F), and then mixed. Flexural test bars were cast from the various mixes, and tests as indicated in tables 3 and 4 were carried out. Test samples similar to the controls were made up except that varying percentages (on a weight basis) of ATC-3 accelerator were added to part B. Part B was made up both with and without the regular uranyl acetylacetone accelerator (UAA).

The following steps were taken:

1. Part B samples (table 3) -- to which varying percentages⁴ of ATC-3 accelerator had been added -- were mixed with part A samples.
2. The following characteristics of the above mixtures were then determined:
 - a. Optimum cure time at 71.1°C (160°F)
 - b. Peak exotherms
 - c. Glass transition (T_g) and heat deflection (HDT) temperatures
 - d. Gel times

¹Product of Cordova Chemical Co, Sacramento, CA.

²1979 dollars.

³Hysol Div, Dexter Corp, Olean, NY.

⁴Based on liquid in part B.

- e. Presence of epoxy in mine sensors and S&A assemblies
 - f. Flexural strengths and moduli
 - g. Compressive strengths and moduli
 - h. Silica filler setting
- i. Stability of part B samples at -28.9°C (-20°F) with and without silica fillers

3. Part B samples were prepared that were similar to those prepared in step 1 but without UAA accelerator (table 4), and characteristics listed in 2 above were determined for these mixtures.

Epoxy cure times were determined with a differential scanning calorimeter. Values obtained were reduced to a unit weight basis, and these data were plotted as functions of time. The cure times are listed in table 5 and the cure time curves are shown in figures 1 through 12.

Normally, the optimum cure times would be determined by the extrapolation of the curves to zero exothermic heat per unit weight. In this case, however, for the purpose of obtaining plateaus and of avoiding abrupt terminations of the curves, zero exothermic heats were not shown. This procedure was followed for the following reasons:

1. The filler which was 77.8 percent of the epoxy could have screened out the effect of residual exothermic heat and simulated a premature ending of the curing reaction.
2. Cure times determined (table 5) from zero exothermic heats proved to be erroneous since the products were either soft or sticky.
3. The presence of ATC-3 accelerator could have prevented some of the anhydrides which are included in part B from reacting, and these could have acted as plasticizer.

Peak exotherms were measured by means of thermocouples that were placed in cups holding freshly mixed epoxy molding compounds. After being filled, the cups were placed in an oven with the temperature held at 71.1°C (160°F). Continuous records were made of the temperatures, and peak exotherms were taken as the temperature increased above 71.1°C (160°F). They are listed in tables 3 and 4.

Gel times (T_g) were determined by manual stirring of the samples and notation of the time when the samples become too viscous for more stirring. This procedure was carried out under a microscope.

Intrusion of the epoxy into mine sensors and S&A assemblies, which is an occasional production problem, was simulated on a laboratory scale. Production molds were used to make housings, which were then cut apart and inspected. Results are given in table 6.

The flexural strength and modulus and the compressive strength and modulus were determined for ATC-3 accelerator mixtures according to ASTM specifications D790 and D695 respectively. Flexural bars were prepared for testing by being conditioned at 50 percent relative humidity and at 22.8°C (73°F) for 88 hours according to ASTM specification D618. Results of tests are shown in tables 3 and 4 and figures 13 through 20.

Glass transition (T_g) and heat deflection (HDT) temperatures were obtained with flexural bars. Glass transition was measured by mechanical expansion as a function of temperature when heated at 10°C (18°F) per minute in a Perkin-Elmer TMA-1 tester. Heat deflection temperatures were the temperatures at which the bars deflected 0.010 inches under a constant load of 264 psi when heated at a constant rate of 2°C (3.6°F) per minute. A Tinius-Olsen heat deflection tester was used to determine heat deflection temperatures. Results are shown in tables 3 and 4.

Settling of silica fibers used as fillers during molding operations was considered a possibility. Cross-sectional specific gravities of flexural bars were determined as listed in table 7 to check this possibility.

The stability of part B with and without fillers and with varying percentages of ATC-3 accelerator was observed after samples placed in glass jars had been conditioned in a cold chamber at -28.9°C (-20°F) for 48 hours. Results are shown in table 8.

Discussion

The ATC-3 accelerator drastically reduces epoxy cure time in proportion to the amount used. The effect is slightly greater when uranyl acetylacetone accelerator is also present. Data in table 5 and figures 1 through 12 illustrate the effect.

A serious loss of strength and toughness exists, both with and without UAA accelerator. Data in tables 3 and 4 and figures 13 through 20 illustrate these effects. All values of strength and toughness are lower than those of the controls, and nearly all are less than required by specification. No test sample, for example, met the flexural strength and flexural modulus requirements of 113 MPa and 18.6 GPa (16,400 psi and 2.7×10^6 psi). Also, no test sample met the compressive strength requirement of 200 MPa (29,000 psi) or the compressive modulus requirement of 15.9 GPa (2.3×10^6 psi).

It is likely that the anhydrides in part B which normally cure the epoxy did not react fully because of the faster reaction of the ATC-3 accelerator with the epoxy. The ATC-3 accelerator contains an organic amine which probably reacts preferentially to the anhydrides. The amine thus leaves some of the anhydrides free to act as a plasticizer in the cured epoxy and thus reduces its physical properties. This conclusion is supported by the work-to-break data in tables 3 and 4. The bars with ATC-3 accelerator did not break because they were too soft, whereas the control bar without ATC-3 accelerator did break because it was hard. Additional curing of a 3 percent ATC-3 bar (table 3) did not change the T_g of 39°C (102.2°F), which indicated that the original degree of cure was complete even though the bar was soft.

The presence of UAA accelerator in the epoxy, along with ATC-3 accelerator, appears to have a rather small effect on physical properties. Only in one case was an increase observed in flexural strength up to 4 percent ATC-3 accelerator in samples having no UAA accelerator in comparison with those containing it. No ready explanation exists for this phenomenon. Figures 17 through 20 illustrate the trends which are tabulated in tables 3 and 4. Again, the general falloff in values may be attributed to the presence of unreacted anhydrides.

Further evidence that unreacted anhydrides were left in the finished products is the relationship of heat deflection temperatures (HDT) to glass transition temperatures (T_g). Heat deflection temperature of the controls were higher than glass transition temperature, but the reverse was true for ATC-3 accelerator samples. Apparently internal changes brought about by ATC-3 accelerator which were reflected by lower gel temperatures were responsible.

Peak exotherms for the ATC-3 accelerator formulations were 3 to 8°C (5.4 to 14.4°F), compared to 3°C (5.4°F) for the controls and 5.6°C (10°F) specification requirements. The differences were small and probably indicate greater degrees of cure, particularly in view of the reduction of gel times from 12 minutes (control value) to much lower values.

Intrusion of epoxy into moving parts proved to be no problem, as indicated in table 6.

Nearly identical specific gravities (table 7) for cross-sections taken from different parts of flexural bars indicated no settling of silica fibers during the molding of ATC-3 samples.

The low temperature stability of the ATC-3 accelerator samples is considered satisfactory since no precipitation was observed (table 8).

PHASE 2 -- ULTRAVIOLET RADIATION OF POLYURETHANE ENCAPSULANT FOR GATOR, GEMSS, AND RAAM MINES

The current method of curing the GATOR, GEMSS, and RAAM mines polyurethane that encapsulates their electronic components consists of exposing the uncured gel at room temperature for 16 hours, followed by oven curing at 71.1°C (160°F) for 2 hours. Keeping the temperature rise or reaction exotherm during this stage at 5.6°C (10°F) or less above the oven temperature is essential since the mine electronics, and especially the batteries, are heat sensitive and can be damaged if heated above 76.7°C (170°F).

Also, a serious encapsulation problem can result from too rapid curing of the polyurethane. High internal stresses develop which cause the plastic to crack and damage the electronics in the assembly. Evidence of this damage is given by an unduly high exotherm. In contrast, slow curing makes possible the relief of internal stresses while the polyurethane is soft, thus preventing cracking and damage to the electronics. A low exotherm correlates with this undamaged condition.

Procedure

The following steps were taken in this investigation:

1. Polyurethane no. 3125 uncured resin mix⁵ samples were cured by ultraviolet radiation and by oven heat.
2. The degrees of cure of ultraviolet-radiated and oven-treated samples were determined by measurement of their physical properties.
3. Temperatures within battery cases (fig. 21) and temperatures of the polyurethane in encapsulated assemblies were measured while these cases and polyurethane were being radiated. Temperatures of polyurethane samples molded to simulate encapsulated assemblies (fig. 22) but without components were also measured while these samples were being radiated. In addition, samples that were oven-cured were prepared for comparison with the samples that had been radiation cured.

In the first step polyurethane test discs were formed as follows:

Polyurethane resin was placed in aluminum foil cups approximately 63.5 mm (2.5 in.) in diameter and 7.6 mm (0.3 in.) deep and was cured in ultraviolet radiation equipment manufactured by the Xenon Corporation⁶. Other polyurethane in similar cups was oven-cured as previously described.

For the second step, specimens for flexural and final hardness testing were machined from the discs described above. Flexural tests were carried out according to ASTM method D790 except for the size of the test bars, which were 57.2 mm long, 12.7 mm wide, and 3.8 mm thick (2.25 in. long, 0.5 in. wide, and 0.15 in. thick). The load span was 25.4 mm (1.0 in.) and the rate of load application was 5.1 mm per minute (0.2 in. per minute). Hardness of the radiated samples was determined 1/2 hour after exposure and after the samples had cooled to room temperature. Hardness of the oven-cured samples was determined only after the samples had cooled to room temperature. Hardness was determined with the D-scale of a Shore durometer.

In the third step, for heat and exotherm measurements, encapsulated assemblies (consisting of battery cases and empty circuit boards) and simulated assemblies (polyurethane only) were formed in standard polycarbonate molds with aluminum back covers. Temperature changes that occurred during the ultraviolet and oven-curing procedures were recorded with a Wahl indicating digital pyrometer. Two temperature probes were inserted in each battery case and circuit board assembly--one within the battery case and one adjacent to it in the polyurethane. One probe was inserted in the simulated electronics assembly.

⁵Product of Hexcel Corp, Chatsworth, CA.

⁶Wilmington, MA.

Discussion

Test results (table 9) for the ultraviolet-radiated samples indicate that higher pulse intensities and higher pulse rates (which produce greater energy outputs) result in higher temperatures, hardness, and flexural strengths than lower pulse intensities and lower pulse rates. The ultraviolet radiation also produced higher temperatures and flexural strengths than the oven cures (table 9). In comparison, the polyurethane exotherm curve (fig. 23) shows temperatures attained when no external heat was provided.

The high temperatures associated with ultraviolet radiation were probably caused by the absorption and reflection of heat from the aluminum back covers on the polycarbonate molds (fig. 22). It is likely, therefore, that the curing process was a thermal one and was not one of photo-initiation (which is catalyzed by ultraviolet radiation), because photo-initiation generally occurs at lower temperatures than were observed in this investigation. The rule that higher temperatures increase reaction rates applies in this case.

The assumption that the polyurethane did not undergo a photo-initiated cure is supported by the fact that the polyurethane does not contain a photo-initiator and none of its normal components react to ultraviolet radiation. Further, because of steric hindrances, the polyurethane when oven-cured contains 1 to 2 percent unreacted isocyanate, and at the higher temperature produced by ultraviolet radiation this material could react, thus increasing the degree of cure.

The similarity of the curves of the oven-cure samples and the ultraviolet-cure samples (fig. 24) also indicates that the ultraviolet cures were thermal and were not photo-initiated. Since the ultraviolet-cure curve is higher than the oven-cure curve, the peak exotherm for the ultraviolet-cure curve is higher and is reached sooner than that of the oven-cure curve. Heating of the samples was stopped when their temperatures reached 60°C (140°F), but their final temperatures went far above the intended maximum of 71.1°C (160°F).

Temperature curves for encapsulated battery case and circuit board assemblies and for simulated assemblies (polyurethane only) show the relative effects of ultraviolet radiation on the polyurethane in each. Again, radiation was stopped at 60°C (140°F). The maximum temperature of the polyurethane in the battery case and circuit board assemblies reached 97.2°C (207°F) after 18 minutes (fig. 25), whereas the maximum temperature of the polyurethane in the simulated assemblies reached 106.1°C (223°F) after 20 minutes (fig. 24). The greater mass of the reacting polyurethane in the latter case was the cause of the higher temperature.

Temperature curves (fig. 25) for battery cases and polyurethane in the encapsulated assemblies show the relative effects of ultraviolet radiation on these components. Early in the radiation period the battery case temperatures reached 63.3°C (146°F), while that of the polyurethane was 60°C (140°F). However, even though radiation was stopped at this point, the polyurethane temperature rose to 97.2°C (207°F), while that of the battery case only went to 81.7°C (179°F). This result indicates that the temperature of an encapsulated battery would be considerably lower than that of the encapsulant and, therefore, the encapsulant exotherm could be quite high without necessarily causing battery damage.

PHASE 3 -- MECHANICAL AND IN SITU BONDING OF GATOR
MINE AND ITS AEROBALLISTIC HOUSING

The GATOR aeroballistic housing and the GATOR mine body can be joined in a single operation in which the body would be used as an insert during molding of the aeroballistic housing. This method of joining the parts would be superior to the present one in which the completed housing is attached to the body by an adhesive. The simultaneous molding and joining of the housing and body would simplify production of the GATOR mine and would make possible significant cost savings. Two methods exist for doing this: (1) mechanical bonding, and (2) in situ bonding.

Mechanical bonding depends upon physical bonding (no adhesive) of the body to the plastic housing. The body would be inserted in the mold in such a manner that the molten plastic could flow into the mold and surround the body. The body would be preheated before it was placed in the mold. Two requirements exist for this type of molding operation to be successful. First, the metal substrate to which the plastic matrix will be joined must be lightly knurled, grooved, or upset to provide a mechanical tooth that will hold the plastic. This toothing could be done without effect on the ballistic performance. Second, the plastic should shrink at least 0.005 in./in. during the molding operation.

A possible advantage of the mechanical bond over the in situ bond is primarily experience with the mechanical bond. Molded-in inserts are common to the trade; many competent molders have had experience with insert mold design and insert molding techniques.

A drawback of mechanical bonding is lack of reliability. The bond between the mine body and the aeroballistic housing would depend primarily upon retention of hoop stress by the housing. Loss of hoop stress would allow the parts to separate since the extent of metal that can be upset without affecting mine ballistic properties is limited. If the aeroballistic housing should crack, the hoop stress would be relieved and the mechanical tooth would not retain the aeroballistic housing when the mine was deployed. Similarly, if the plastic housing should relieve the hoop stress by creep, the mechanical bond again would be significantly weakened. Temperature extremes could cause both cracking and creep.

Mechanical bonding might be satisfactory if a mine body metal upset consisting of a 0.64 to 0.76 mm (0.025 to 0.030 in.) circumferential groove were provided. Loading on the aeroballistic housing would have to be determined to make sure of this. The mine body might collapse in the mold if the pressure of the plastic in the mold was too high. If this pressure were a problem, additional tooling would be required to support the mine body.

The estimated cost of mechanical bonding is based primarily on information furnished by Engineered Plastics Company⁷ (table 10). The mold now used for producing the aeroballistic housing could be modified to accept the body as an

⁷Philadelphia, PA.

insert for \$2000.⁸ Such a mold would cost about \$15,000 new, and 3 months would be required to make it. When tooling was available, Engineered Plastics could mold 50,000 assemblies per month at a price of \$0.70 to \$0.80 each. Engineered Plastics could also provide limited metal working facilities for light knurling or for machining grooves in the outer surface of the body. For smaller runs of 2000 assemblies per month, with use of a material such as a nylon at \$1.64/lb, the total cost would include tooling as described, plus \$1.60 for each assembly.

In situ bonding requires adhesion between the plastic and the metal part. For this operation, an adhesive is usually applied to a metal part before it is inserted in the mold cavity. Contact between the molten plastic and the adhesive causes the parts to adhere to each other. Usually, a longer molding cycle and a slightly higher holding temperature is used than in mechanical bonding to increase flow, wetting, and coupling of the molten plastic and the adhesive. Also, the coated metal part may be preheated before it is placed in the mold, and the completed assembly may be subjected to a post-anneal treatment. Metal upset may also be provided but is not essential.

The advantage of the in situ bonding is its high reliability, since the adhesive and mechanical bonds together provide two fastening techniques in one assembly. With adhesive bonding, intimate contact occurs between the surfaces of the metal insert and the plastic matrix, thus making stress distribution more uniform. If the plastic encapsulant should crack, little change in load bearing characteristics would occur, since hoop stress in the plastic does not maintain the bond by itself.

According to information received from Springborn Laboratories, Inc.,⁹ present design of the aeroballistic housing and the housing mold so the mold will accept the mine body as an insert (table 10). The cost of a new mold that would include the present core would be \$15,000. A completely new mold with associated parts would cost between \$20,000 and \$25,000. With the necessary tooling available, small runs with different plastics could be made at a cost of \$2.50 to \$3.00 per assembly in lots of 1000. With production quantities of 50,000 assemblies, costs would be \$2.00 per assembly for a single cavity mold and \$0.65 to \$0.75 per assembly for a four-cavity mold.

Information from Engineered Plastics in regard to in situ bonding indicated that the length of the molding cycle would have to be doubled and that this plus coating of the mine body would increase the cost of each assembly to \$1.20 at a production rate of 50,000 assemblies per month.

Both of the above molding companies are confident that the mine body and aeroballistic housing can be joined in a single operation and they both agreed that a metal upset would be required for mechanical fastening alone. Since the

⁸All estimates are in 1979 dollars.

⁹Enfield, CT.

metal upset could alter the fragmentation pattern and aeroballistic characteristics of the mine, the effect of such a modification should be determined by testing in the field.

Either of the above molding procedures would cost less than the present practice of separately molding the aeroballistic housing and attaching it to the mine body with an adhesive. By this procedure, each assembly now costs \$11.13. However, it has been learned from Iowa Army Ammunition Plant (which loads GATOR, GEMSS, and RAAM mines) that conversion to an in situ bonding method would not be practical at this time. Too much tooling used in common for all three mines would have to be modified.

CONCLUSIONS

Phase 1

1. Addition of ATC-3 accelerator to the ADAM mine epoxy molding compound reduced cure time drastically.
2. Physical properties of the epoxy were reduced below acceptable limits by ATC-3 accelerator.
3. A residue of unreacted anhydrides in the cured epoxy was probably the cause of the reduced physical properties.
4. The presence of an organic amine in the ATC-3 accelerator, that reacted preferentially to the anhydrides, was the likely cause of the residue of unreacted anhydrides in the final product.
5. The addition of ATC-3 accelerator to part B of the ADAM epoxy did not affect its stability, did not cause intrusion of epoxy into moving parts, and did not result in the settling of silica fibers.

Phase 2

1. A thermal reaction and not a photo-initiated reaction took place in the polyurethane samples subjected to ultraviolet radiation.
2. The high temperatures produced by ultraviolet radiation resulted in higher flexural strengths than the lower oven-cure temperatures. However, the increase in strength is not considered significant.

RECOMMENDATIONS

Phase 1

Continue using the present epoxy formulation without change with the ADAM mine.

Phase 2

1. Continue the current practice of first curing the polyurethane at room temperature and then oven-curing it at 71.1°C (160°F) for 2 hours. This procedure will ensure proper encapsulation of mine electronics.
2. In any future investigation of polyurethane curing methods, the effects of higher exotherms and ultraviolet radiation should be further explored.

Phase 3

An in situ bonding capability for the GATOR mine and its aeroballistic housing should be considered for any facility built in the future for production of GATOR, GEMSS, and RAAM mines. A reduction in cost from \$11.13 to \$3.00 or less per GATOR assembly is presently indicated, and a comparable reduction should be realized at a later date.

REFERENCES

1. Kuo Chang, "Determine the Effect of Cordova ATC-3 Accelerator for Improving Properties and Cure Time of Molding Compound for ADAM A/P Mines, Project 155MM, M692/M731," Contractor Report, Defense Systems Division, Honeywell, Inc., Hopkins, MN, February 1980.
2. J. Gulbierz and R. Foster, "U.V. Flash Polymerization Study on a Polyurethane Resin Designated Hexel #3125," Laboratory Report 299, Materials and Manufacturing Technology Division, FC&SCWSL, ARRADCOM, Dover, NJ, June 1980.
3. W. C. Tanner, "Feasibility of Molding and Joining the Aeroballistic Housing and Case or Case-Liner Assembly of the GATOR Mine," Laboratory Report 79 LCA-OA-4, Materials and Manufacturing Technology Division, FC&SCWSL, ARRADCOM, Dover, NJ, November 1979.

Table 1. Ingredients furnished by Hysol for epoxy molding compound used with ADAM mine

<u>Hysol's designated number</u>	<u>Ingredient description</u>
--	Uranyl acetylacetone
IM0322 filler	Silica filler (silica sand)
NB509-104	Anhydride eutectic mixture (Tazzle) plus dipropylene glycol (no uranyl acetylacetone)
AS-111 part A	Part A
AS-111 part B	Part B

Table 2. Composition of epoxy molding compound
for use with ADAM mine^c

<u>Ingredient</u>	<u>Percent by weight</u>
<u>Base Resin Composition (Part A)</u>	
Epoxy resin	17.39
2,3 epoxy propanol	3.07
Gamma-glycidoxypropyltrimethoxy silane	1.37
Granular silica sand	53.20
Powdered silica sand	24.96
Foam reducer	0.01
<u>Hardener Composition (Part B)</u>	
Maleic anhydride ^a	4.80
Chlorendix anhydride ^a	6.41
Methyl tetrahydrophthalic anhydride ^a	9.59
Dipropylene glycol ^b	0.41
Uranyl acetylacetone ^b	0.24
Gamma-glycidoxypropyltrimethoxy silane	1.35
Granular silica sand	52.40
Powdered silica sand	24.78
Foam reducer	0.01

^a Anhydrides blended to form a liquid trianhydride eutectic mixture, commonly referred to as "Tazzle".

^b These two ingredients blended to form a solution, then incorporated into and reacted with the trianhydride eutectic mixture.

^c Per Military Specification 48245A.

Table 3. Physical properties of standard epoxy used with ADAM mine including ATC-3 accelerator and UAA accelerator

Item	Flexural strength MPa psi		Flexural modulus GPa 10 ⁶ psi		Work to break sample J/m in.-lb/in. ²		Compressive strength Rbs psi		Compressive moduli GPa 10 ⁶ psi		T _g °C °F		HDT °C °F		Gel time min.	
	Spec reqs	to	113.1	16,400	18.6	2.70	--	200.0	29,000	15.9	2.30	70	158.0	6f	10.8f	
Control	137.2	to	163.4	23,700	24.8	3.60	to	263.0	38,200	26.2	to	3.80	161-min-	20e	—	
2% ATC-3 (std cure) ^a	47.8	to	19,900	19.8	2.87	5,914	33.77	210.0	30,400	19.0	2.76	61.	161.8	75	167.0	
2% ATC-3 (inter- rupted cure) ^b	35.7	to	5,180	7.5	1.09	8,945	>51.08d	51.7	7,500	5.7	0.83	37	98.6	31	87.8	
3% ATC-3 (std cure)	34.1	to	4,940	6.3	0.92	9,660	>55.16d	53.8	7,800	7.1	1.03	39	102.2	31	87.8	
4% ATC-3	30.8	to	4,460	7.0	1.01	8,294	>7.36d	51.4	7,450	5.4	0.79	37	98.6	—	—	
5% ATC-3	34.3	to	4,980	5.5	0.79	9,103	>51.98d	43.9	6,370	5.9	0.85	38	100.4	30	86.0	
6% ATC-3	23.8	to	3,445	4.8	0.69	5,907	>33.73d	34.5	5,010	3.7	0.54	35	95.0	25	77.0	
3% ATC-3 (additional cure after demolding) ^c	39.0	to	5,655	8.1	1.18	613	.3.5	78.6	11,400	11.1	1.61	39	102.2	—	—	

^a These data were obtained from confirmation test which received straight 16 hours cure at 71°C (160°F).

^b Cured for 8 hours at 71°C (160°F); then postcured an additional 8 hours after attempt to demold failed.

^c Postcured an additional 39 hours outside of the mold.

^d Flexural bar did not break; therefore the numbers are meaningless.

^e At 121°C (250°F).

^f At 71°C (160°F).

Table 4. Physical properties of standard epoxy used with ADAM mine,
including ATC-3 accelerator and without UAA accelerator

Item	Plexural strength <u>Mpa</u> <u>psi</u>	Flexural modulus <u>GPa</u> <u>10⁶ psi</u>	Work to break sample <u>J/m²</u> <u>in.-lb/in.²</u>	Compressive strength <u>Mpa</u> <u>psi</u>	Compressive modulus of elast <u>GPa</u> <u>10⁹ psi</u>	T _B <u>°C</u> <u>°F</u>	HDT <u>°C</u> <u>°F</u>	Gel time <u>min.</u>	Peak exotherm <u>°C</u> <u>°F</u>									
2% ATC-3	44.0	6,391	8.6	1.25	3,000	17.13	65.4	9,480	9.2	1.34	40	104.	31	87.8	7	6	10.8	
3% ATC-3	46.7	6,783	9.2	1.34	2,716	15.51	71.7	10,393	9.3	1.35	37	98.6	34	93.2	6	6	10.8	
4% ATC-3	48.4	7,020	8.2	1.19	3,805	21.73	65.3	9,470	8.3	1.20	37	98.6	32	89.6	4	7	12.6	
5% ATC-3	38.6	5,603	6.1	0.89	No break ^a		55.8	8,090	6.9	1.00	37	98.6	34	93.2	3	6	10.8	
6% ATC-3 (std cure)	23.9	3,470	5.2	0.75	No break ^b		50.1	7,272	6.1	0.88	37	98.6	29	84.2	2	8	14.4	
0.75% ATC-3	40.4	5,855	10.9	1.58	1,406	8.03	93.3	13,530	12.2	1.77	39	102.2	--	--	--	11	3	5.4
6% ATC-3 ^c (longB cure)	47.5	6,890	13.9	2.01	1,010	5.77	99.6	14,440	13.6	1.97	44	111.2	--	--	--	--	--	--

^a Bar did not break; tested at 5,012 J/m² (28.62 in. lb/in.²).

^b Bar did not break; tested at 9,993 J/m² (57.06 in. lb/in.²).

^c Received 21 hr of cure time instead of 6 hr cure time.

Table 5. Cure times at 71.1°C (160°F) of the standard epoxy used with ADAM mine, including ATC-3 accelerator (with and without UAA accelerator)^a

<u>Percent of ATC-3</u>		<u>Planned cure time (hr)</u>
<u>With UAA</u>		
0		40 ^a
2		16
3		12 ^b
4		10
5		8
6		6
<u>Without UAA</u>		
0.75		30
2		21
3		19
4		16
5		10
6		6

^a 40-hour cure was based on MIL-M-48245A (military specification "Housing and Plug") control characteristics.

^b Actual cure time should be 12 hours (fig. 3). The 3% ATC-3 epoxy was cured for 16 hours to insure that parts could be removed from the mold readily.

Table 6. Epoxy intrusion tests

<u>Percent of ATC-3</u>	<u>Sensor</u>							<u>S&A mechanism</u>
	<u>1</u>	<u>2</u>	<u>3</u>	<u>4</u>	<u>5</u>	<u>6</u>	<u>7</u>	
<u>With UAA</u>								
Control	N	N	N	N	N	N	N	N
2	N	N	N	N	N	N	N	N
3	N	N	N	N	I	N	N	N
4	N	N	N	N	N	N	N	N
5	N	I	N	N	N	N	N	N
6	N	N	N	N	N	N	N	N
<u>Without UAA</u>								
2	N	N	N	N	N	N	N	N
3	N	N	N	N	N	N	N	N
4	N	N	I	N	N	N	N	N
5	N	N	N	N	N	N	N	N
6	N	N	N	N	N	N	N	N

N Indicates negative intrusion.

I Indicates slight positive intrusion, but in an amount too small to cause malfunction of sensor.

Table 7. Filler settling based on specific gravity of the cast bars*

<u>Percent of ATC-3</u>	<u>Upper end spec gravity</u>	<u>Lower end spec gravity</u>	<u>Diff in spec gravity</u>
<u>With UAA</u>			
Control	2.17	2.17	0
2	2.16	2.16	0
3	2.16	2.16	0
4	2.16	2.16	0
5	2.15	2.15	0
6	2.15	2.15	0
<u>Without UAA</u>			
2	2.15	2.16	-0.01
3	2.15	2.15	0
4	2.15	2.15	0
5	2.15	2.15	0
6	2.15	2.15	0
0.75	2.16	2.16	0

* Bars cast in vertical position.

Table 8. Low temperature^a stability studies^{b,c}

<u>Percent of ATC-3</u>	<u>Condition</u>
<u>Part B without filler (silica fibers)</u>	
0	Partially-frozen (approximately 90% crystallized) ^d
2	Partially-frozen (approximately 90% crystallized) ^c
3	Partially-frozen (approximately 50% crystallized) ^c
4	Thickened (0% crystallized)
5	Thickened (0% crystallized)
6	Thickened (0% crystallized)
<u>Part B with filler (silica fibers)</u>	
0	Very stiff (0% crystallized)
2	Very stiff (0% crystallized)
3	Very stiff (0% crystallized)
4	Stiff (0% crystallized)
5	Stiff (0% crystallized)
6	Moderately-stiff (0% crystallized)

^a Low temperature = -28.9°C (-20°F).

^b No precipitation was observed for all of these test samples after they warmed to room temperature. This observation was continued for more than one week.

^c Refer to tables 1 and 3.

^d Visually estimated.

Table 9. Properties of ultraviolet-radiated polyurethane

Specimen no.	Pulse per sec.	Energy (joules/pulse)	Pulse duration (sec.)	Temp °C °F	Initial hardness (D-scale of Shore durometer)	Flexural strength MPa psi	Final hardness (D-scale of Shore durometer)*	
							mm	in.
1	7	288	6	121.1 250	70	106.9 15,500	1.42 0.056	72
2	7	200	6	91.1 196	60	104.5 15,160	1.45 0.057	76
3	7	128	6	93.3 200	55	105.8 15,340	1.73 0.058	76
4	6	288	6	121.1 250	60	109.8 15,930	1.37 0.054	73
5	6	200	6	91.1 196	40	103.1 14,950	1.85 0.073	72
6	5	288	6	87.8 190	40	99.5 14,440	1.68 0.066	74
7	5	200	9	85.6 186	55	100.9 14,640	1.32 0.052	75
8	5	200	9.5	88.3 191	60	97.1 14,080	1.32 0.052	74
9	5	200	12	86.1 187	60	104.6 15,170	1.55 0.061	75
10	5	128	12	75.6 168	40	92.8 13,460	1.91 0.075	76
Control	--	--	--	71.1 160	--	95.8 13,890	1.32 0.052	74

* Hardness reading taken 1/2 hour after removal from heat source.

Table 10. Estimated costs for mechanical and in situ bonding*

<u>Item</u>	<u>Engineered Plastics Co.</u>	<u>Springborn Laboratories, Inc.</u>
Modification of present mold	\$2,000	\$5,000
New mold plus present core	--	\$10,000 to \$15,000
Mechanical bonding; 50,000 assemblies		
4-cavity mold	\$0.70 to \$0.80/assy	\$0.65 to \$0.75/assy
1-cavity mold	\$1.20/assy	\$2.00/assy
Mechanical bonding; 1000-2000 assemblies		
1-cavity mold	\$1.60/assy	\$2.50-\$3.00/assy
In situ bonding; 50,000 assemblies		
4-cavity mold	\$1.20/assy	--

* 1979 dollars.

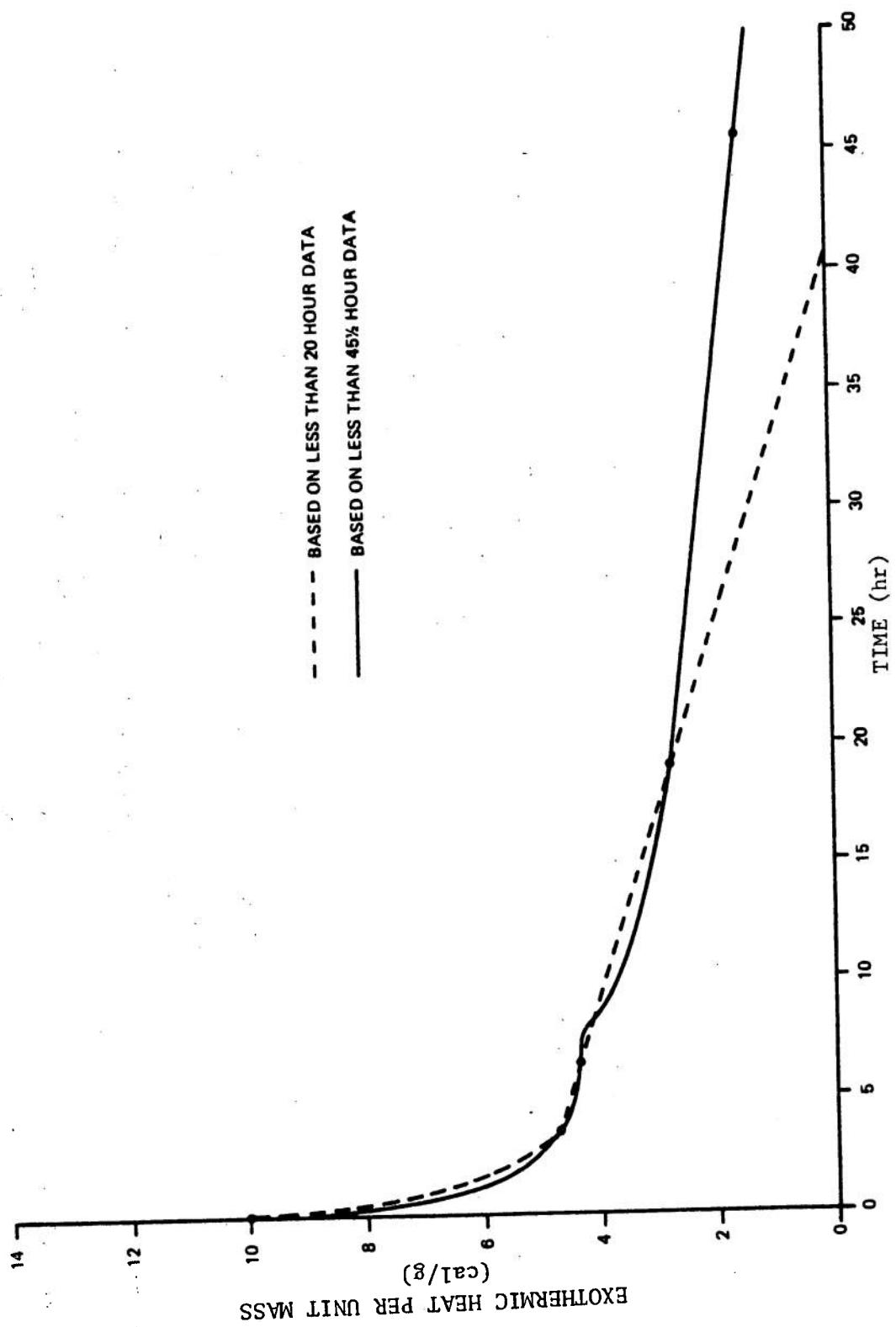
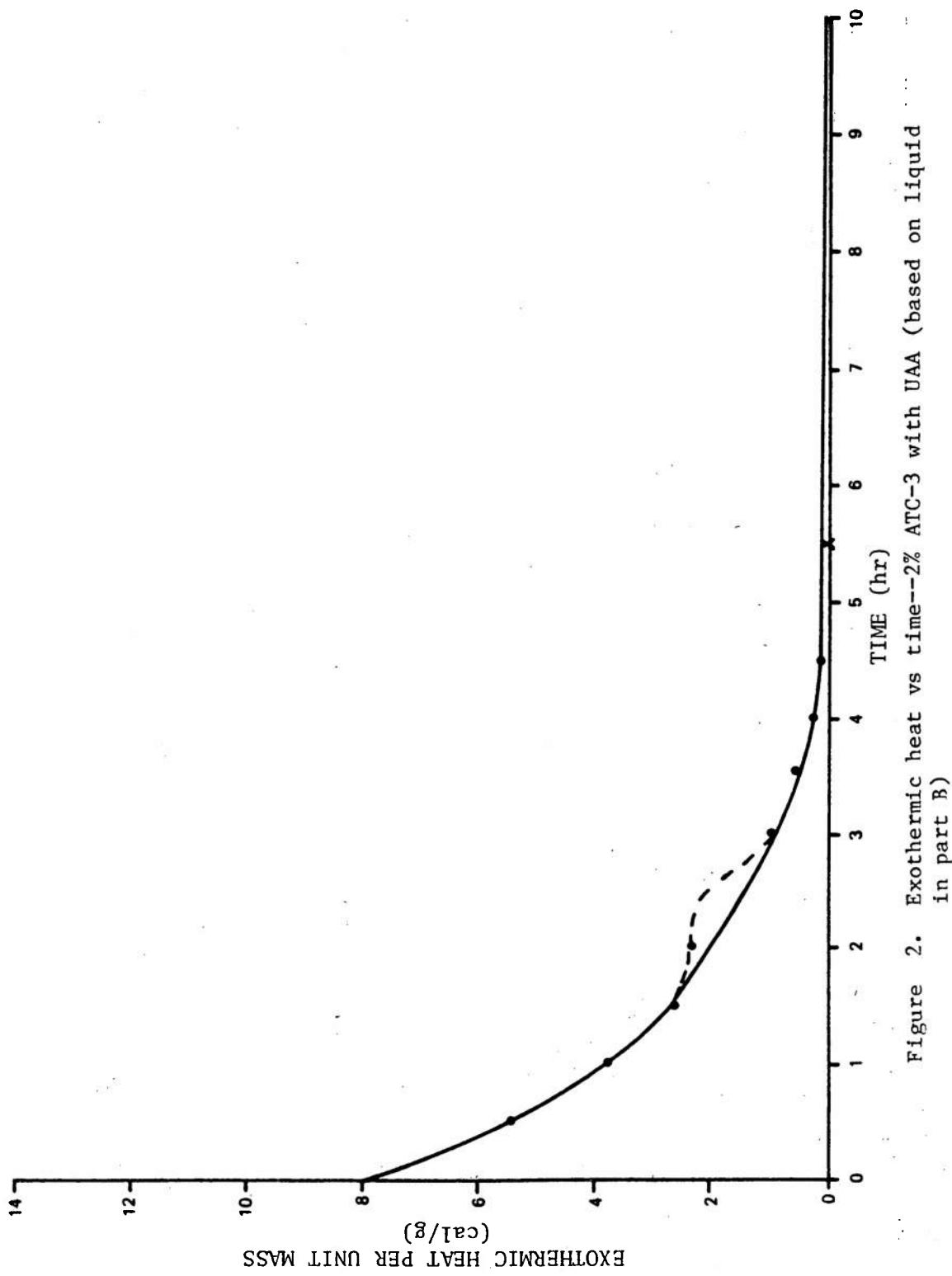


Figure 1. Exothermic heat vs time--control with UAA and without ATC-3



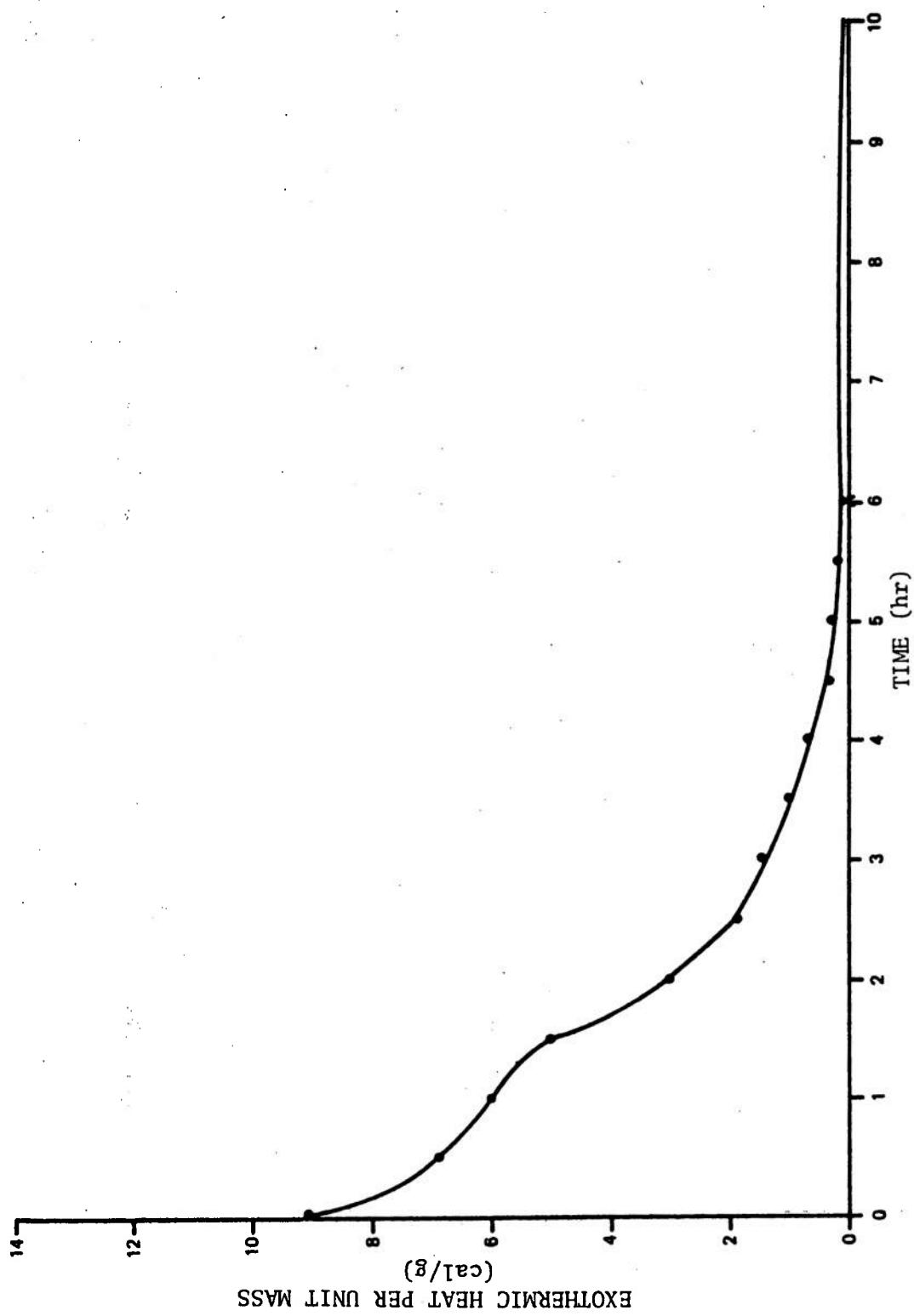


Figure 3. Exothermic heat vs time--3% ATC-3 with UAA (based on liquid in part B)

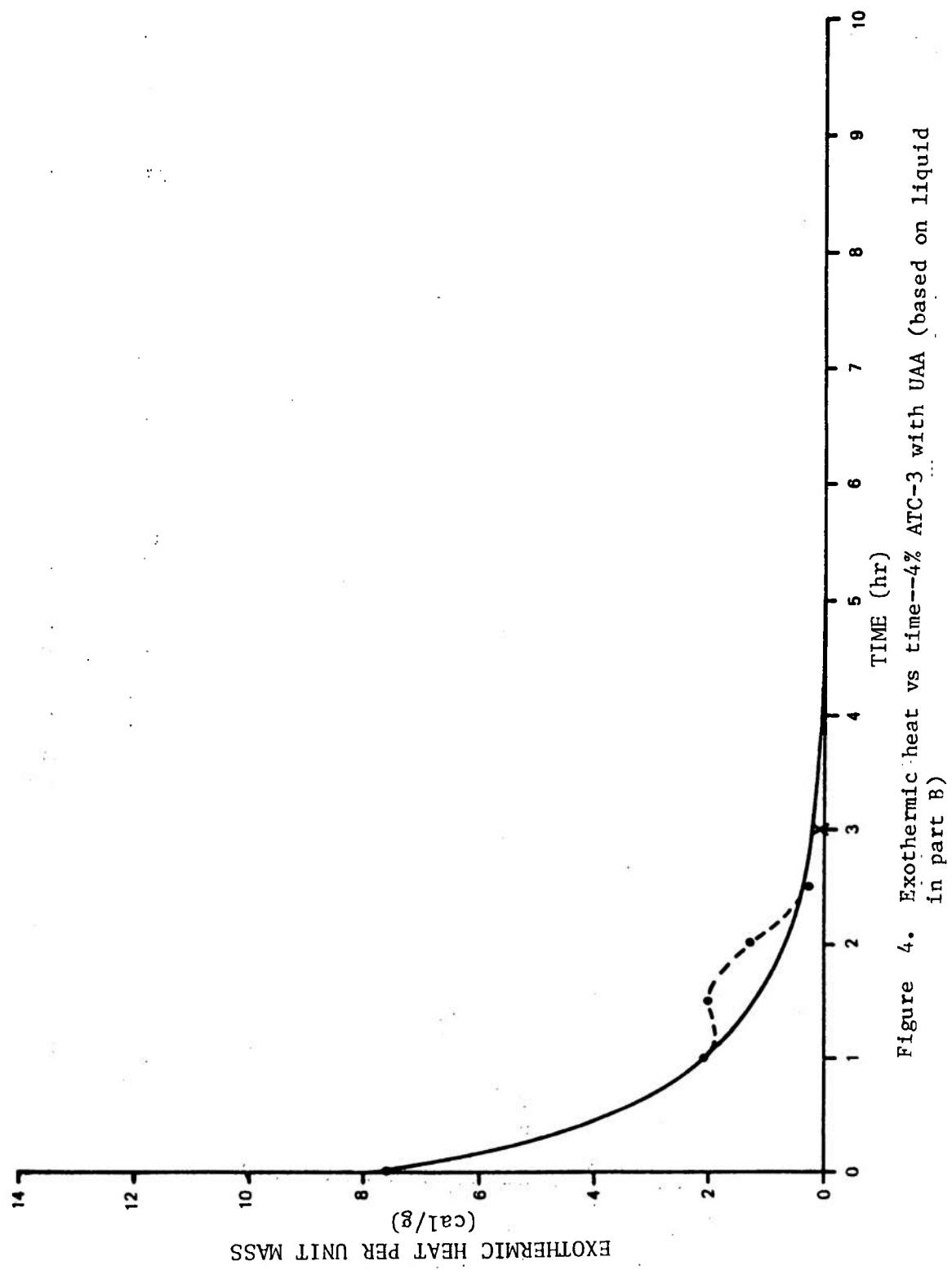


Figure 4. Exothermic heat vs time--4% ATC-3 with UAA (based on liquid in part B)

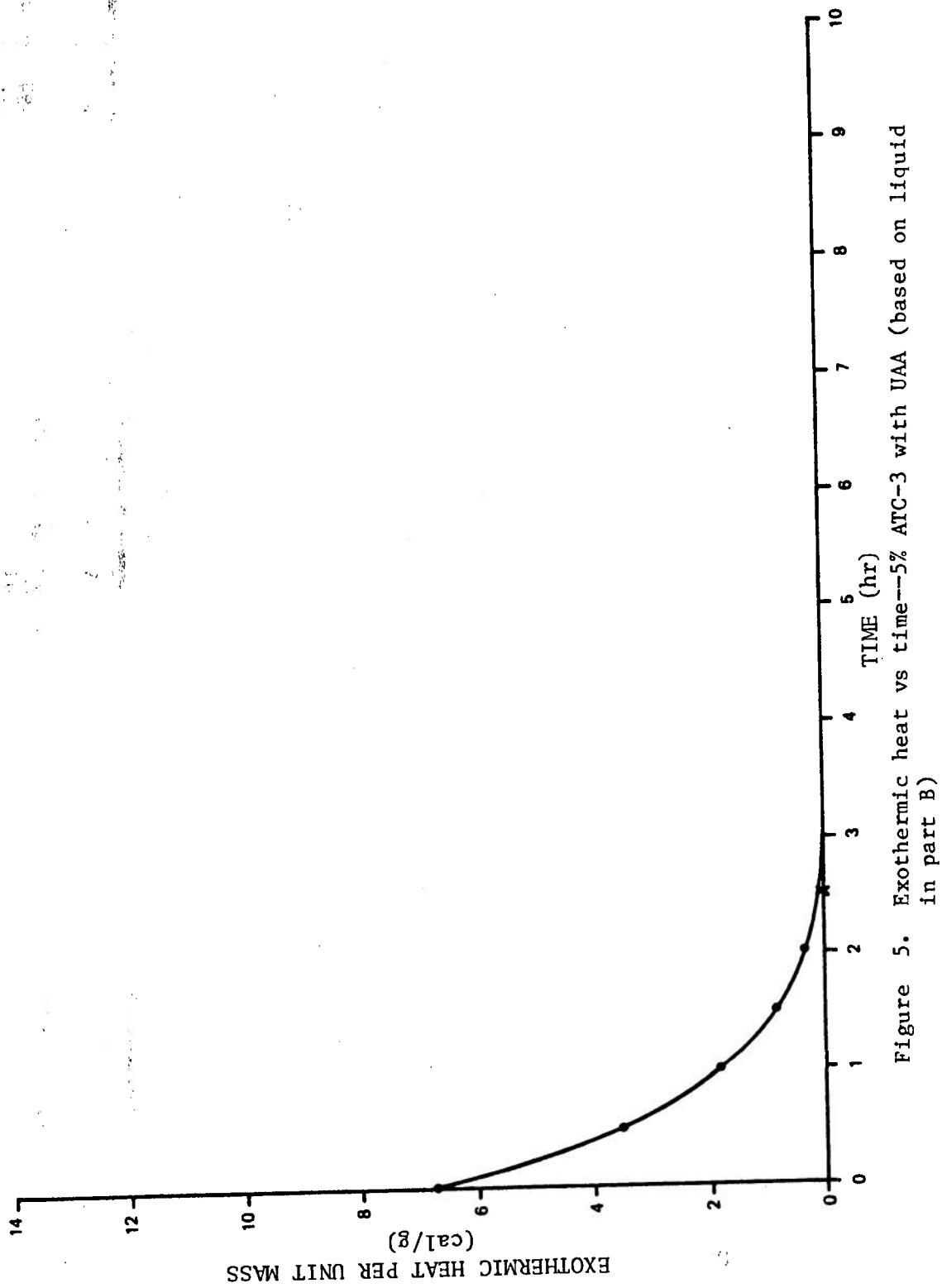


Figure 5. Exothermic heat vs time—5% ATC-3 with UAA (based on liquid in part B)

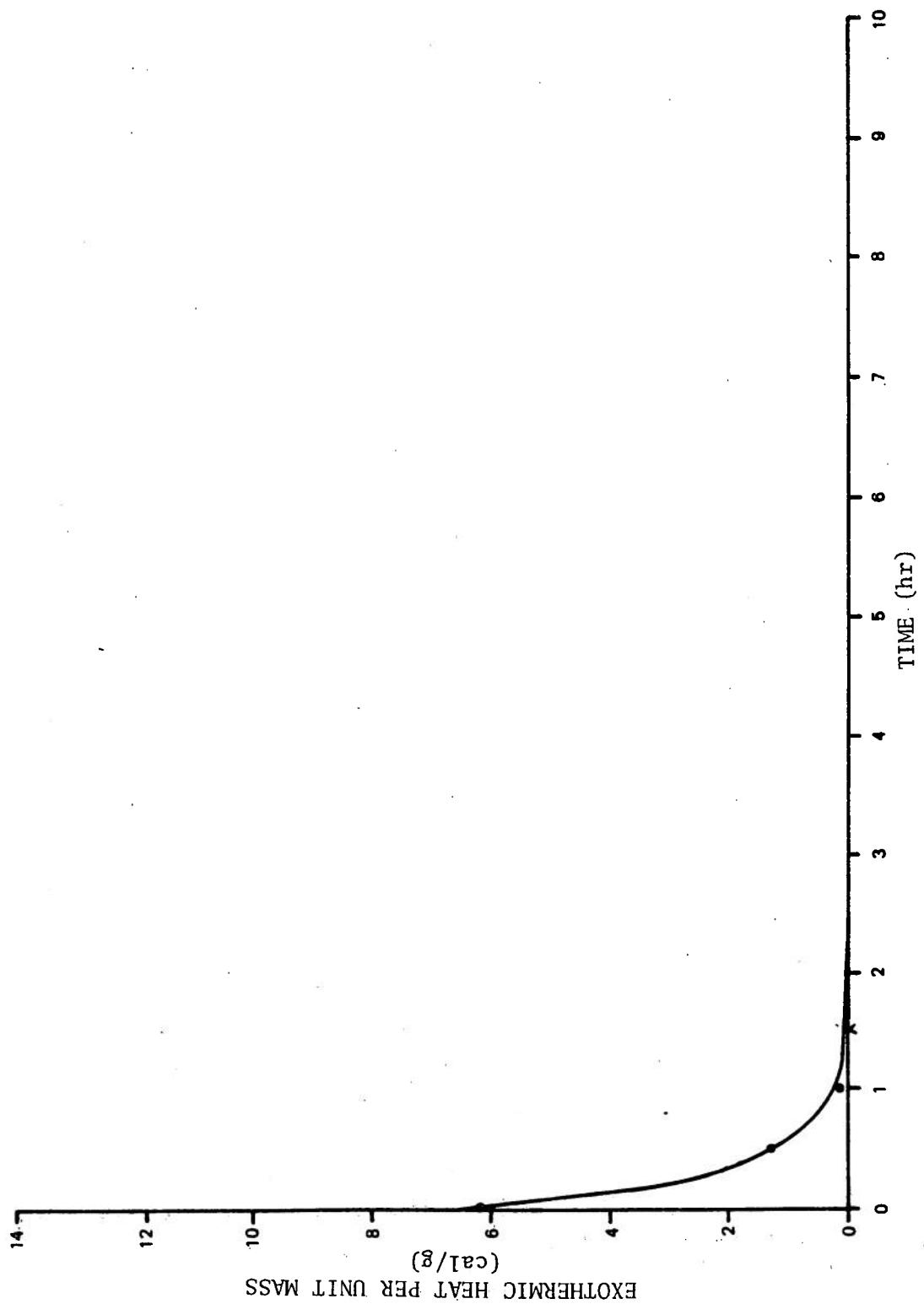


Figure 6. Exothermic heat vs time--6% ATC-3 with UAA (based on liquid in part B)

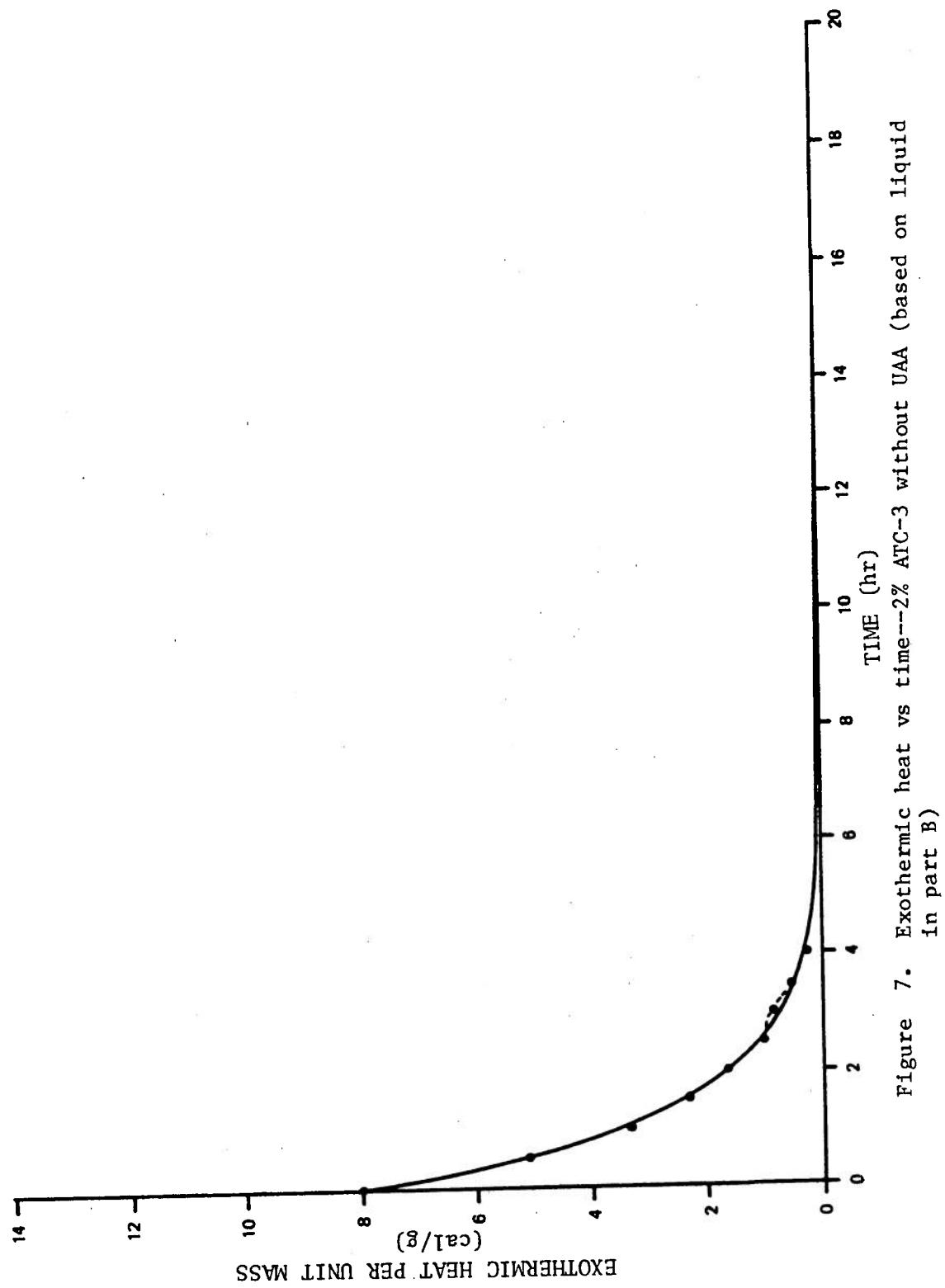


Figure 7. Exothermic heat vs time--2% ATC-3 without UAA (based on liquid in part B)

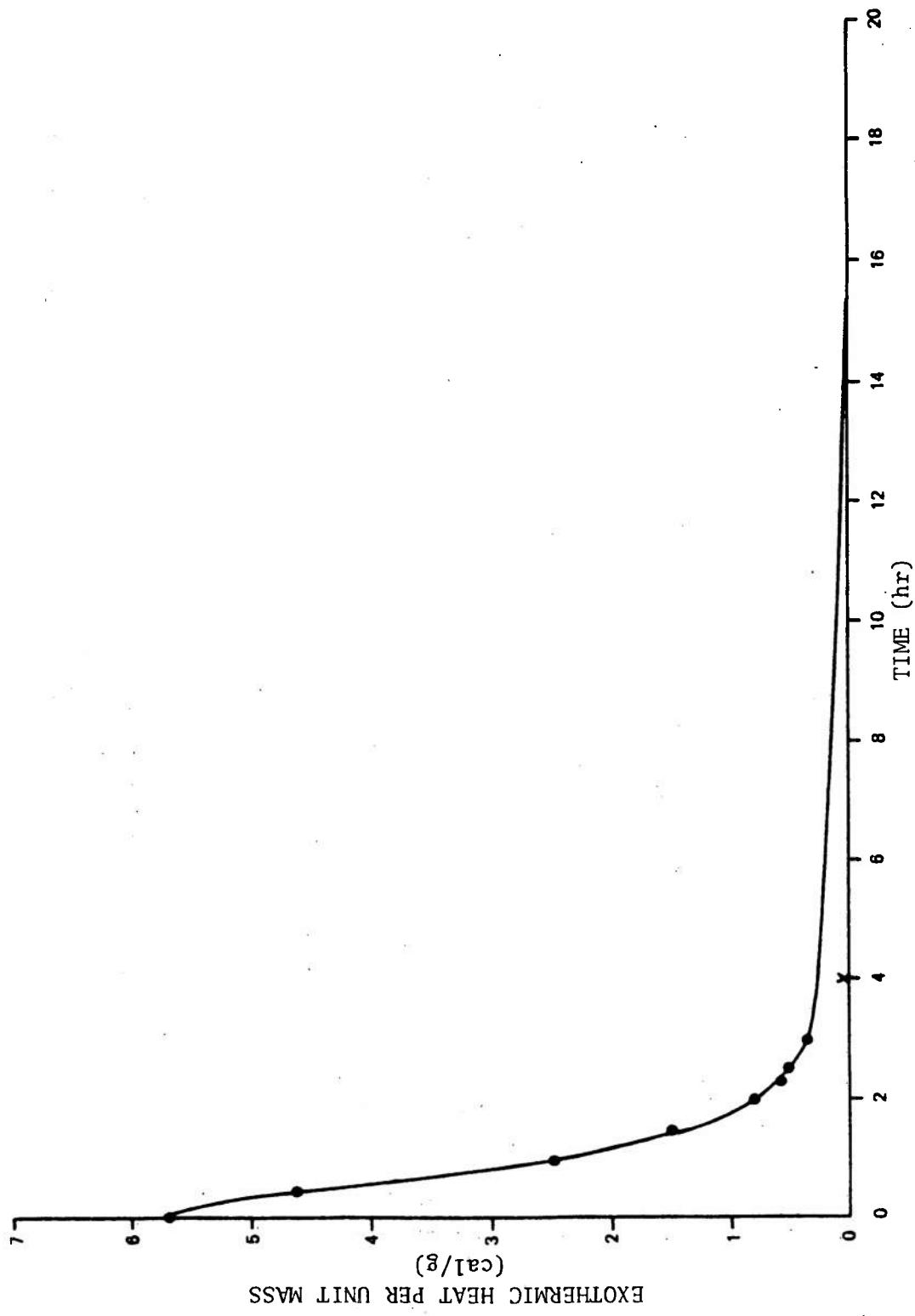


Figure 8. Exothermic heat vs time--3% ATC-3 without UAA (based on liquid in part B)

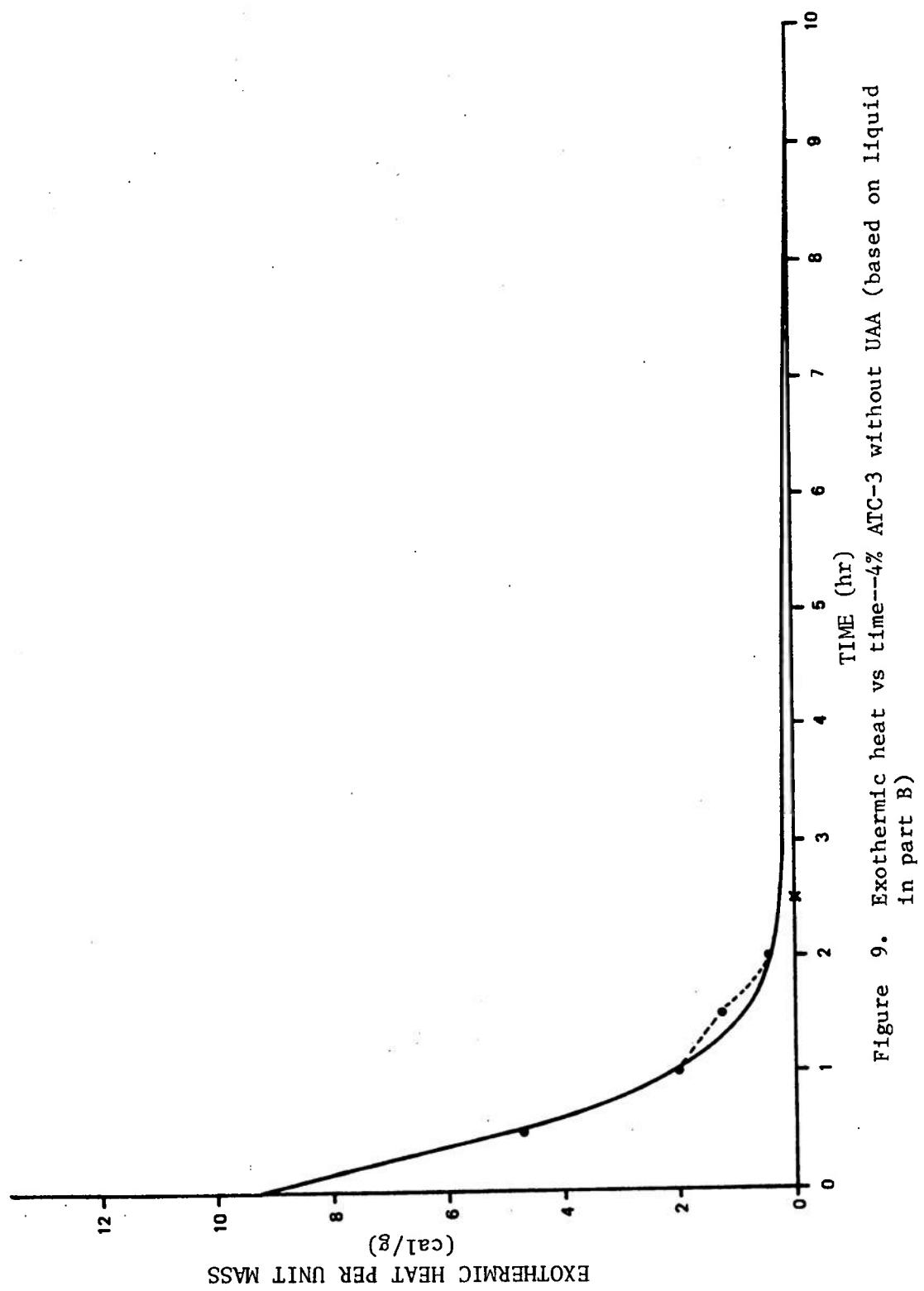


Figure 9. Exothermic heat vs time--4% ATC-3 without UAA (based on liquid in part B)

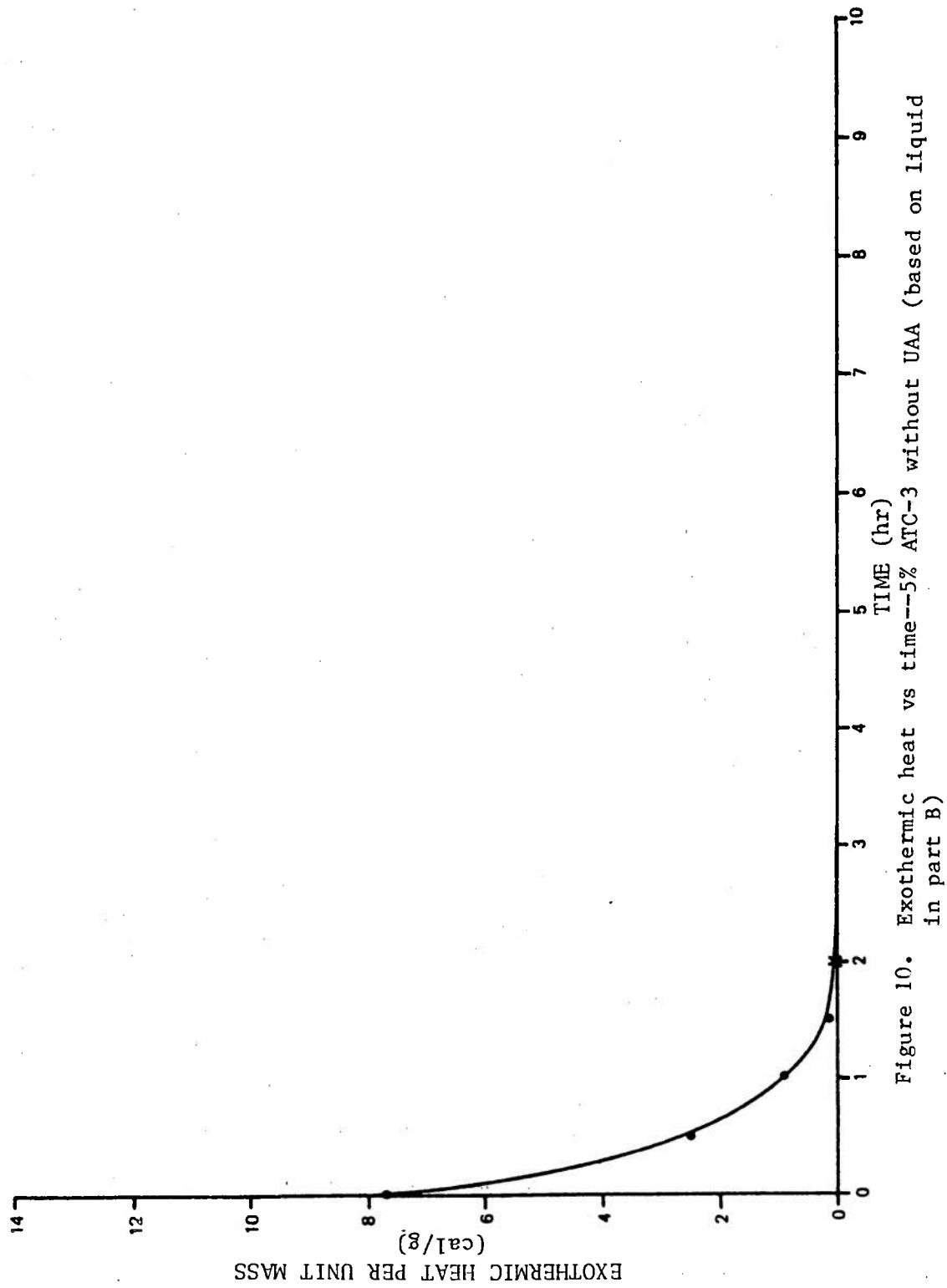


Figure 10. Exothermic heat vs time--5% AFC-3 without UAA (based on liquid in part B)

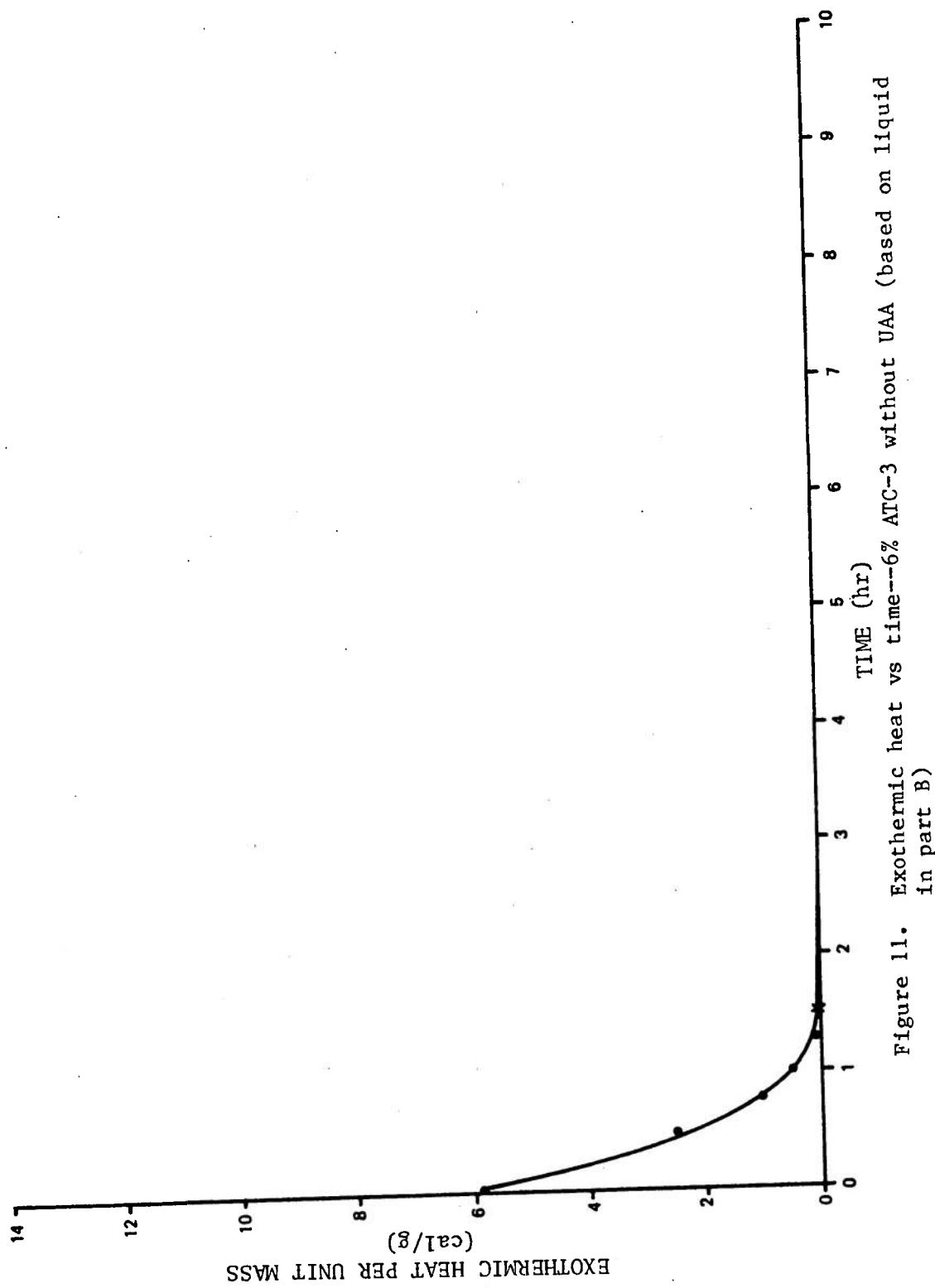


Figure 11. Exothermic heat vs time—6% ATC-3 without UAA (based on liquid in part B)

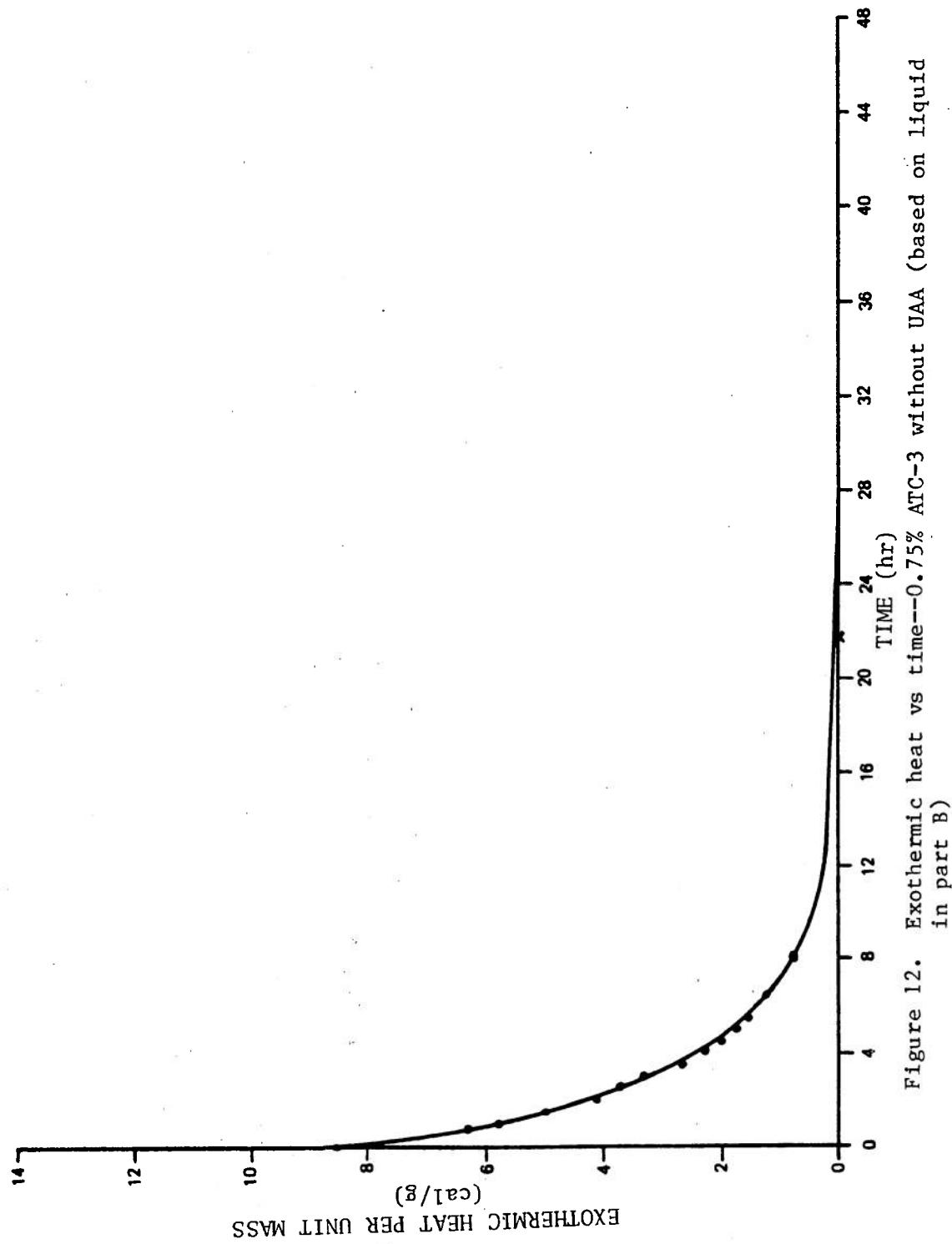


Figure 12. Exothermic heat vs time--0.75% ATC-3 without UAA (based on liquid in part B)

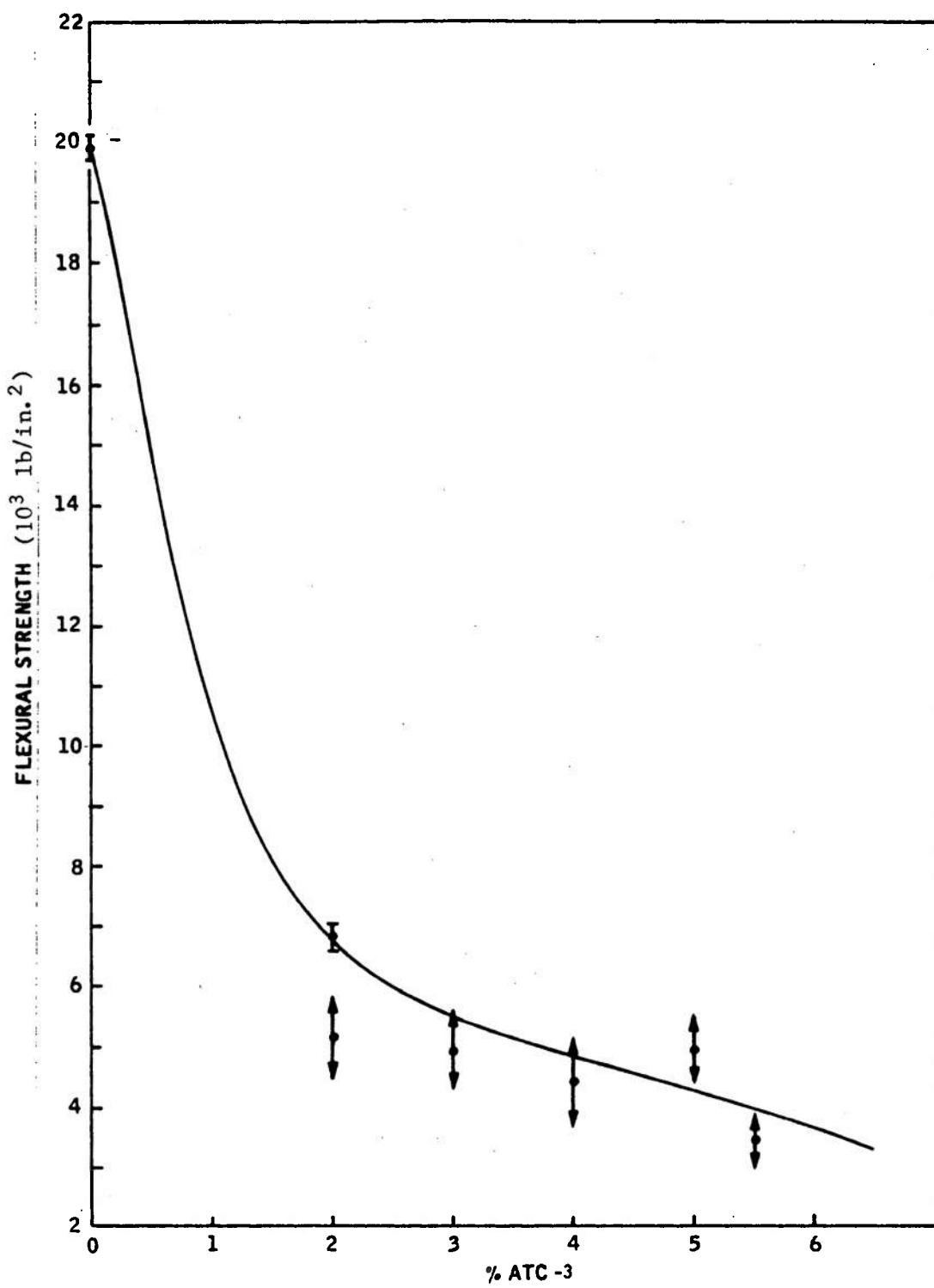


Figure 13. Flexural strength vs ATC-3 content in ADAM mine epoxy with UAA

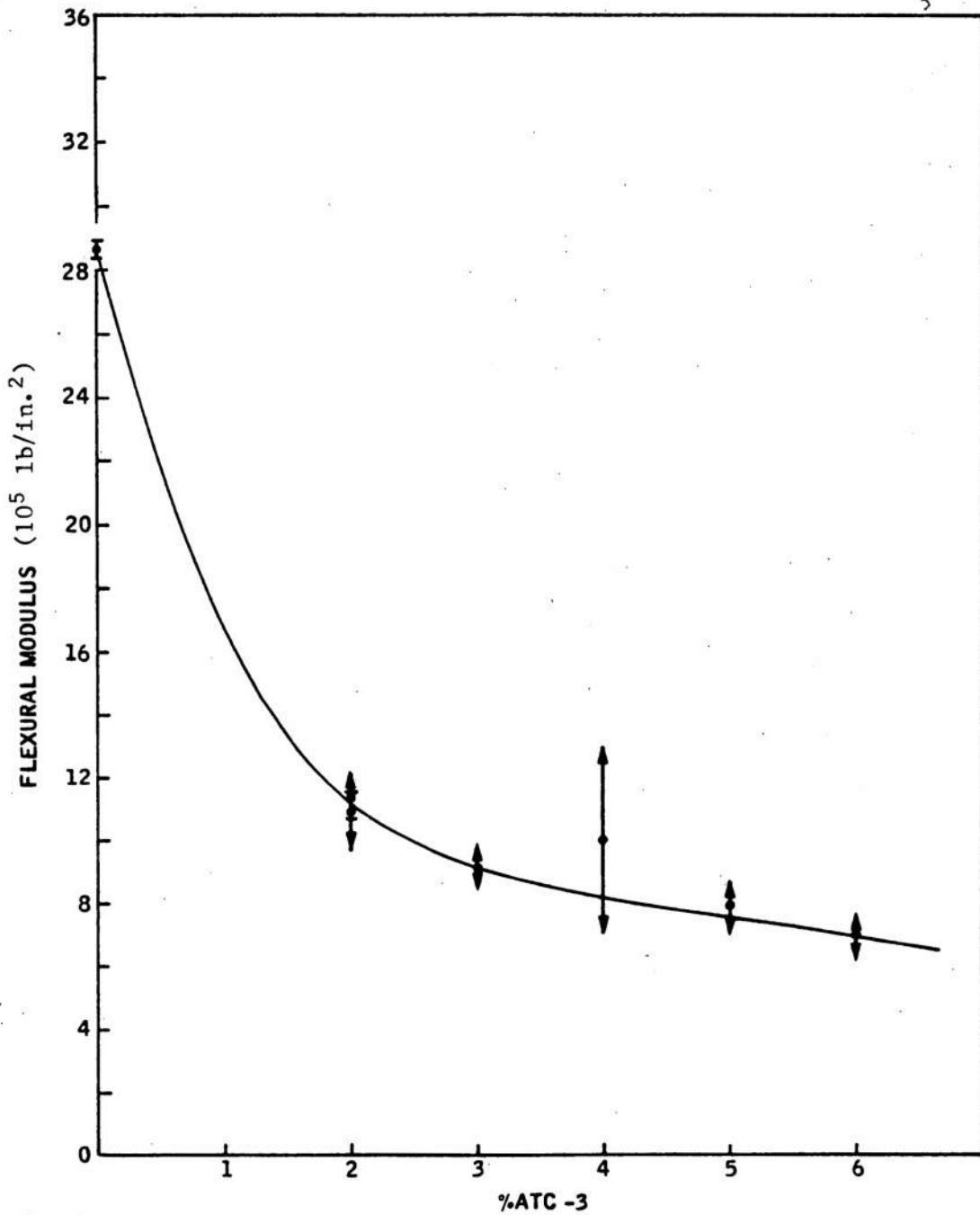


Figure 14. Flexural modulus vs ATC-3 content in ADAM mine epoxy with UAA

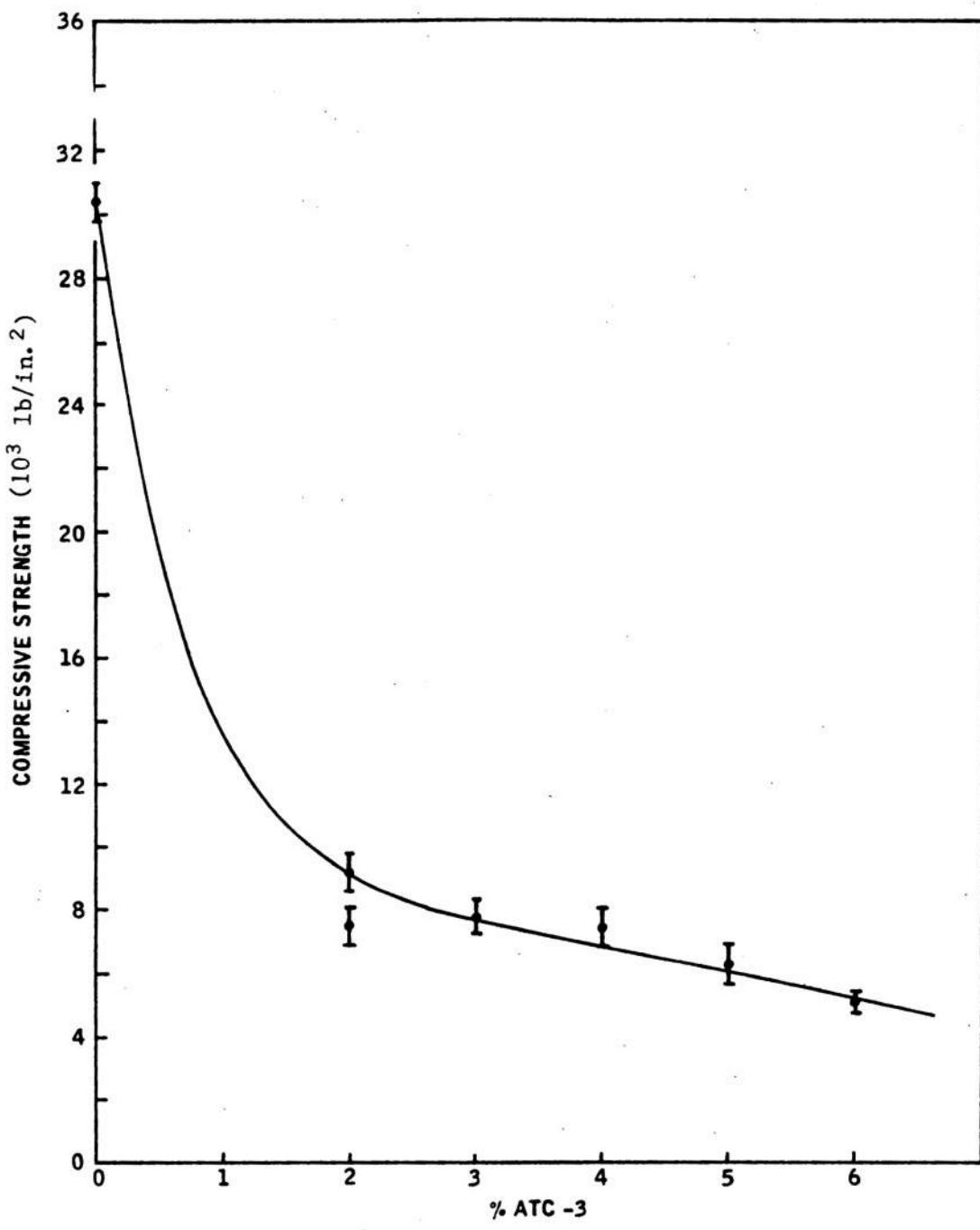


Figure 15. Compressive strength vs ATC-3 content in ADAM mine epoxy with UAA

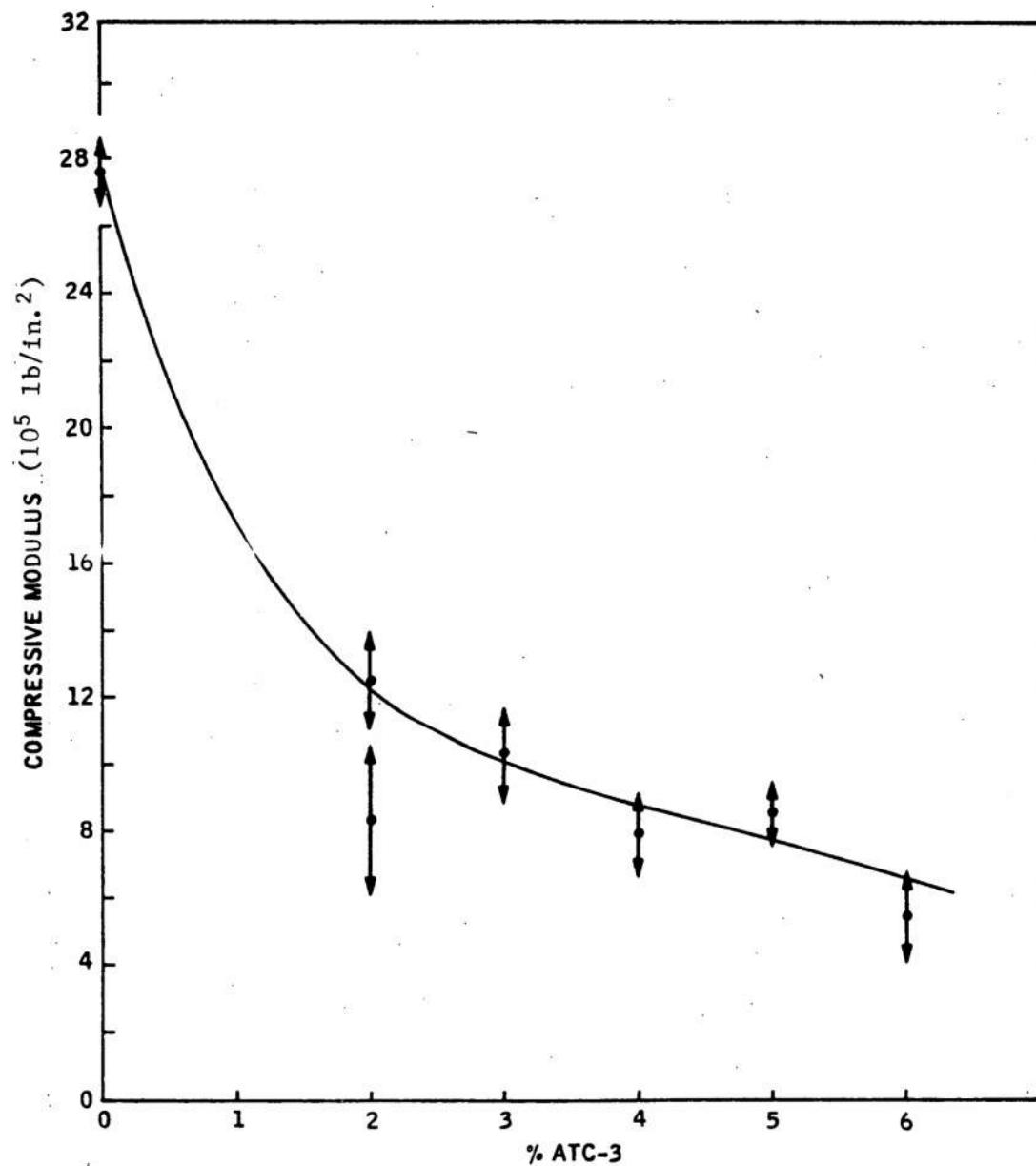


Figure 16. Compressive modulus vs ATC-3 content in ADAM mine epoxy with UAA

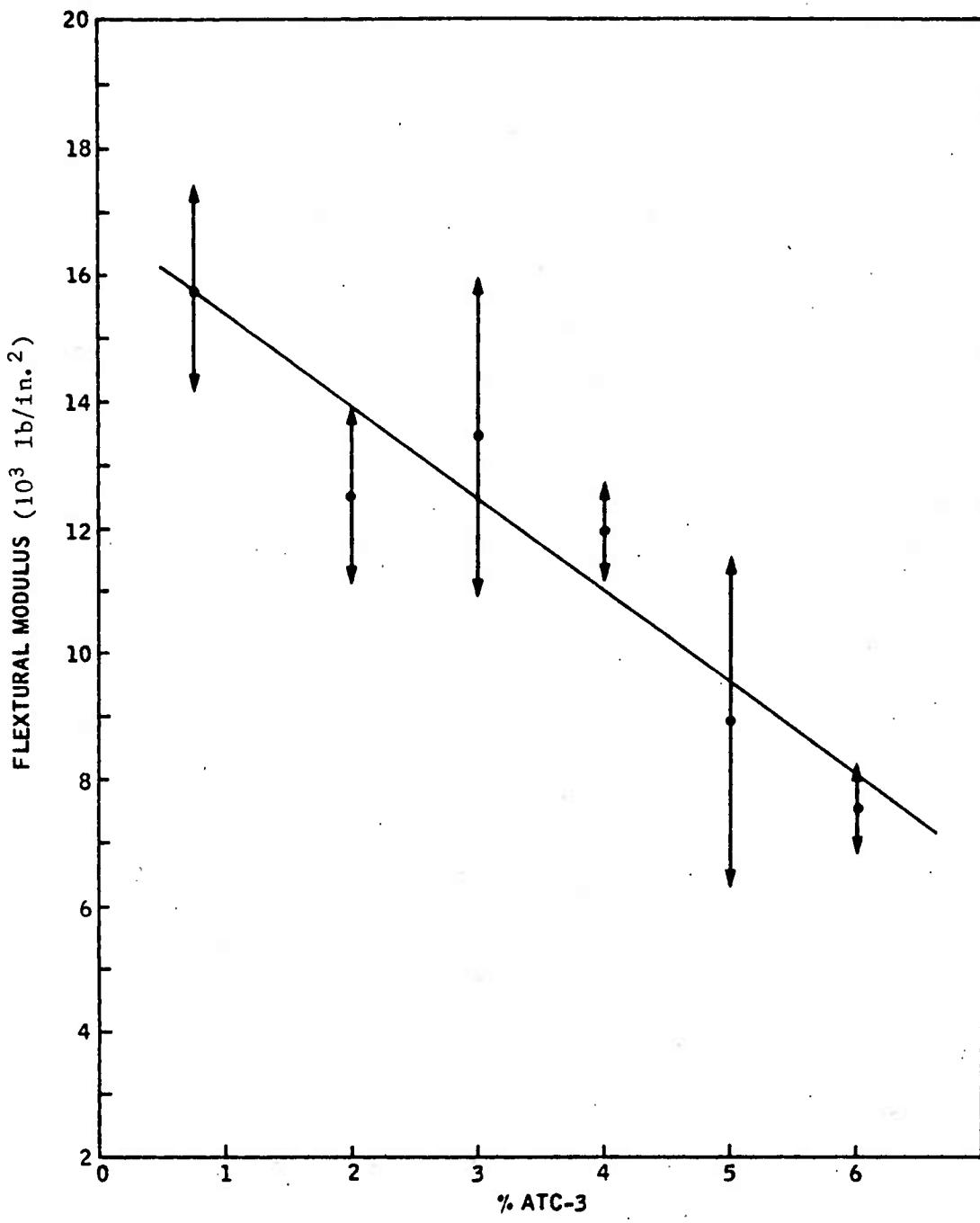


Figure 17. Flexural strength vs ATC-3 content in ADAM mine epoxy without UAA

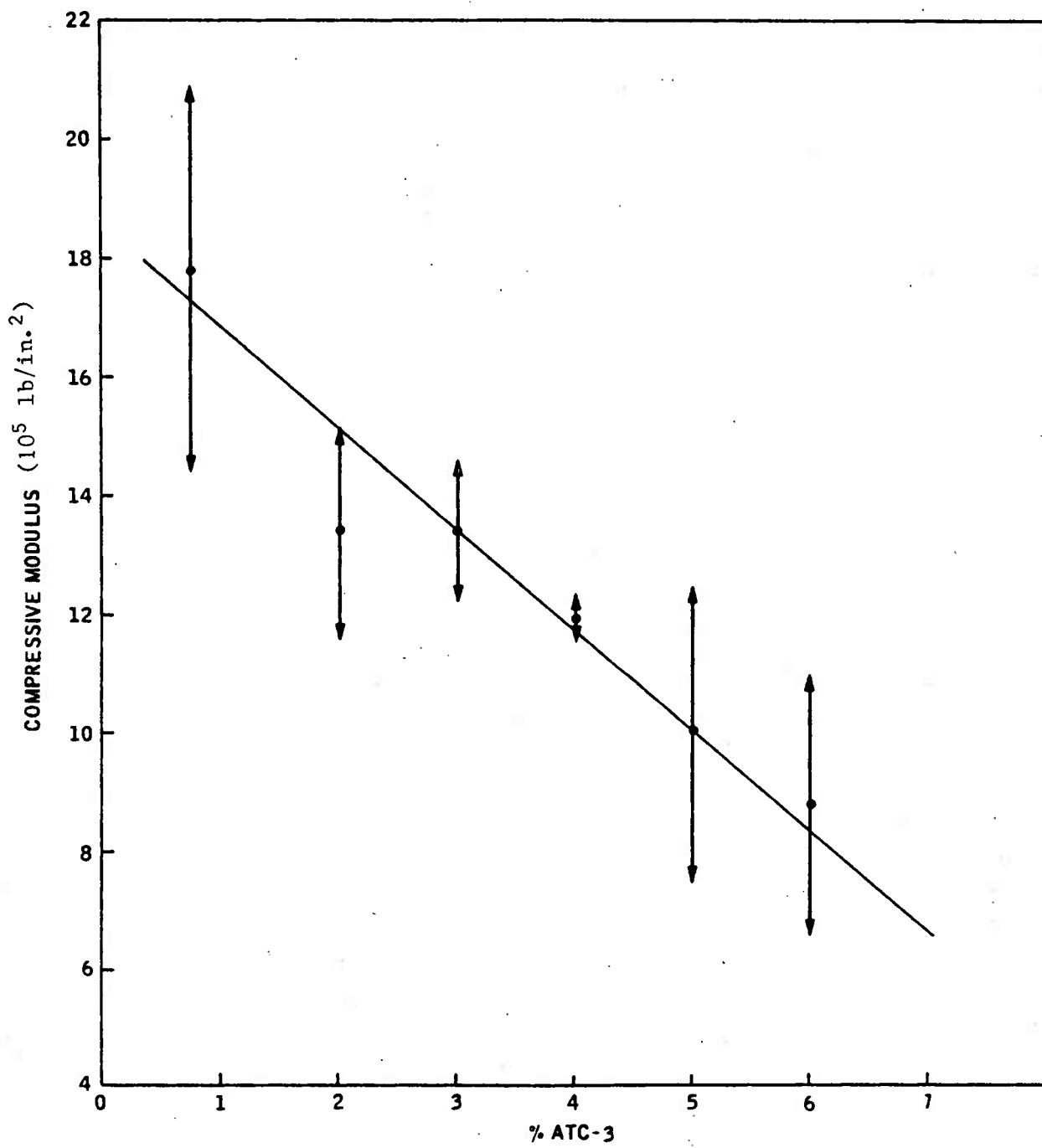


Figure 18. Flexural modulus vs ATC-3 content in ADAM mine epoxy without UAA

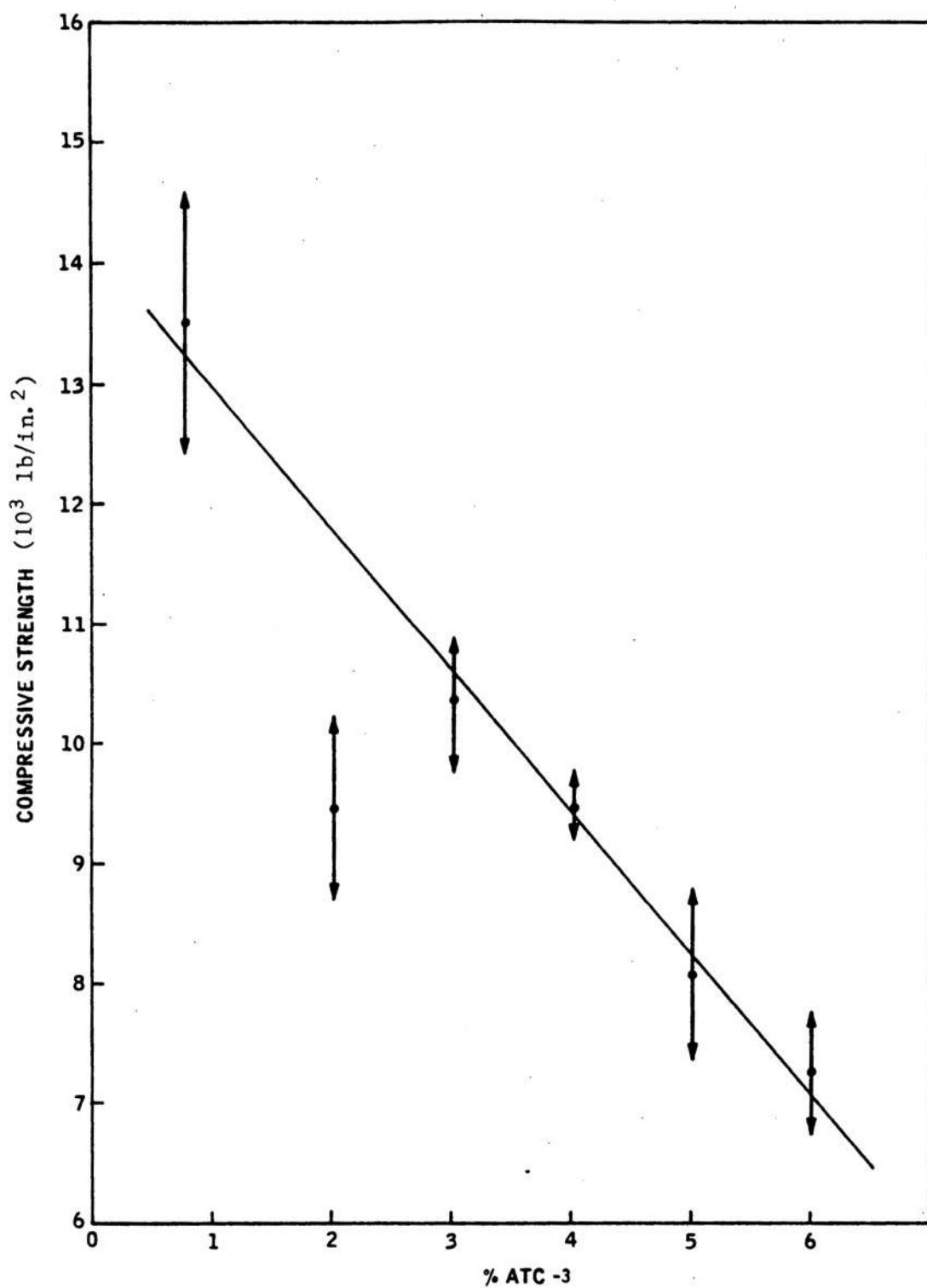


Figure 19. Compressive strength vs ATC-3 content in ADAM mine epoxy without UAA

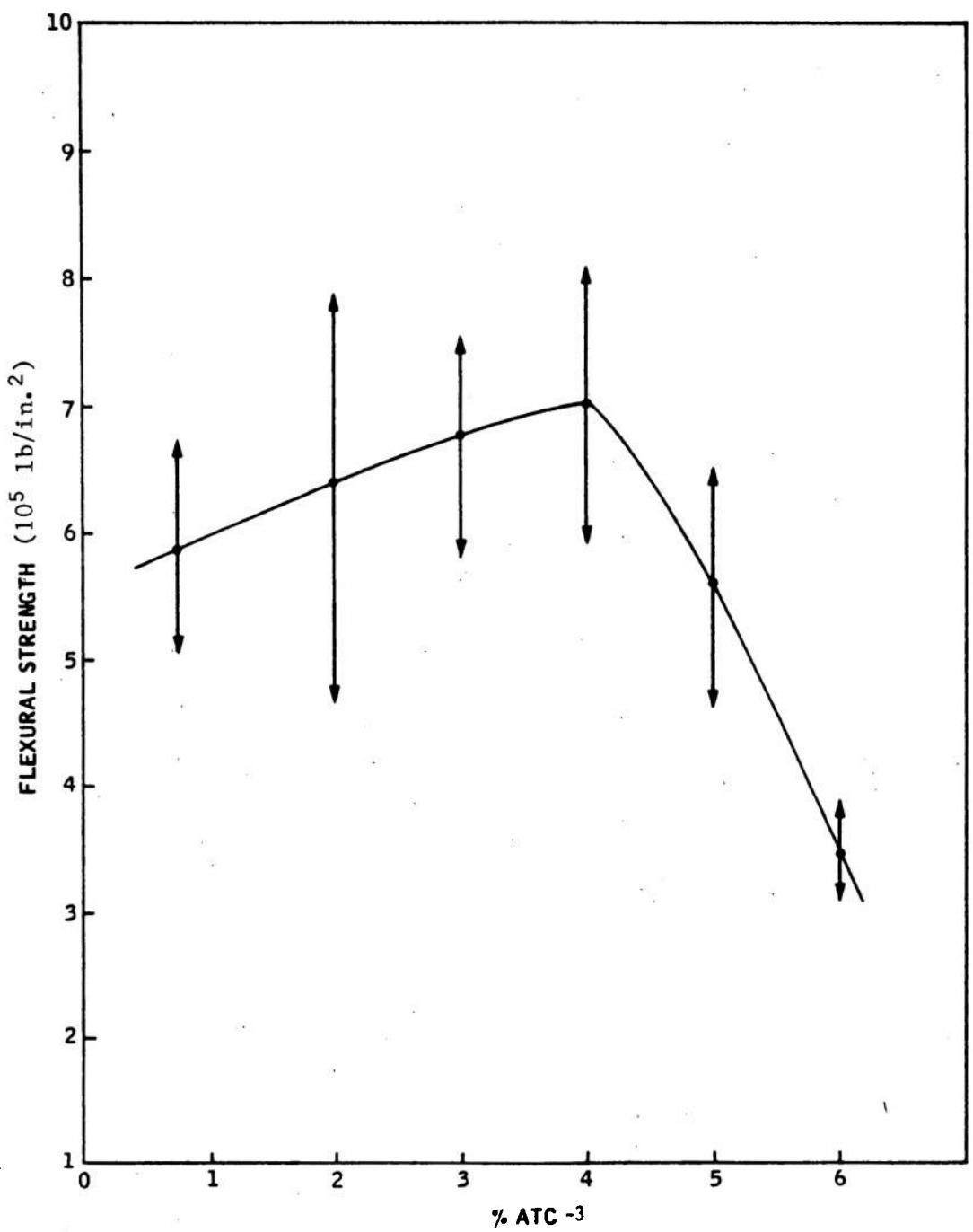


Figure 20. Compressive modulus vs ATC-3 content in ADAM mine epoxy without UAA

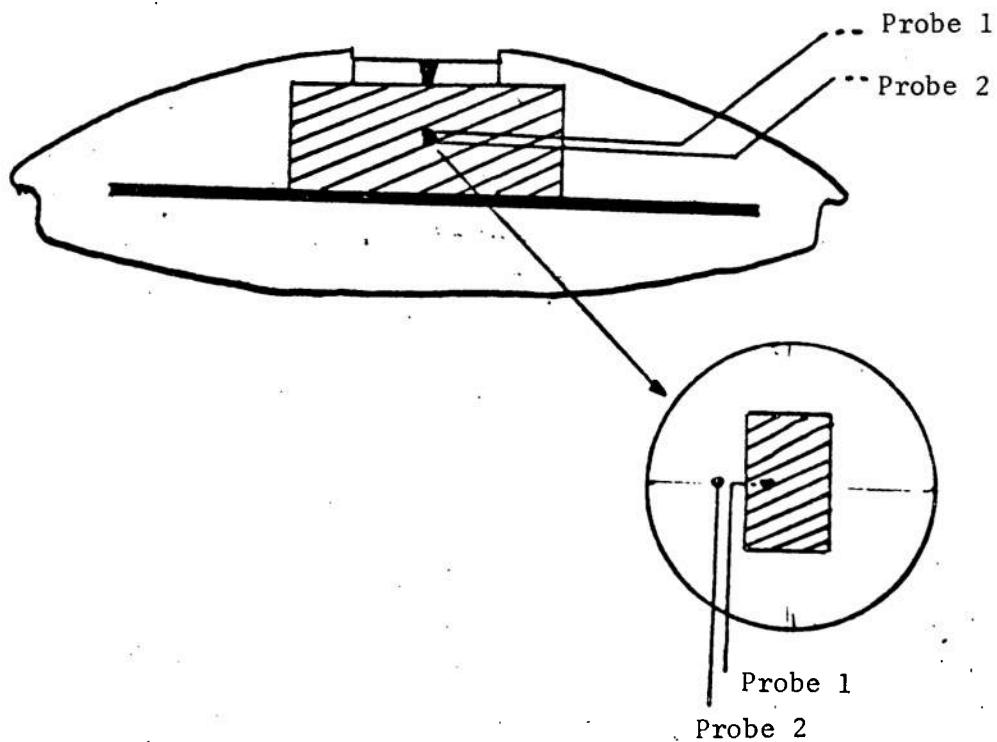


Figure 21. Encapsulated assembly with battery case and with circuit board

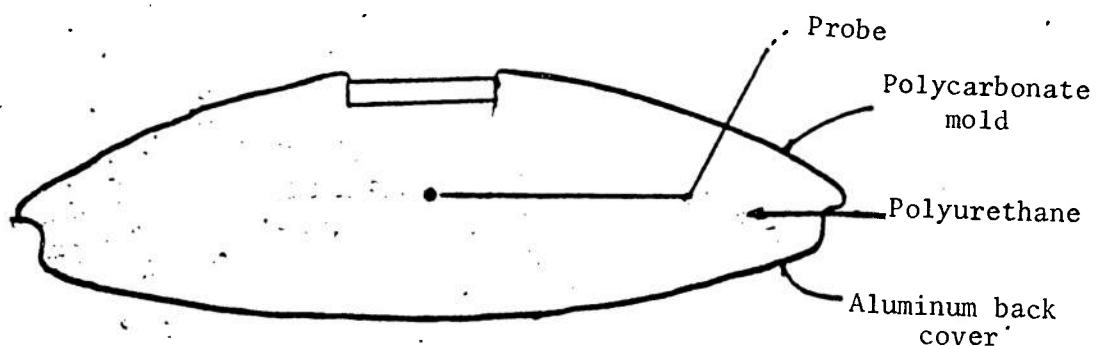


Figure 22. Encapsulated assembly without battery case and without circuit board

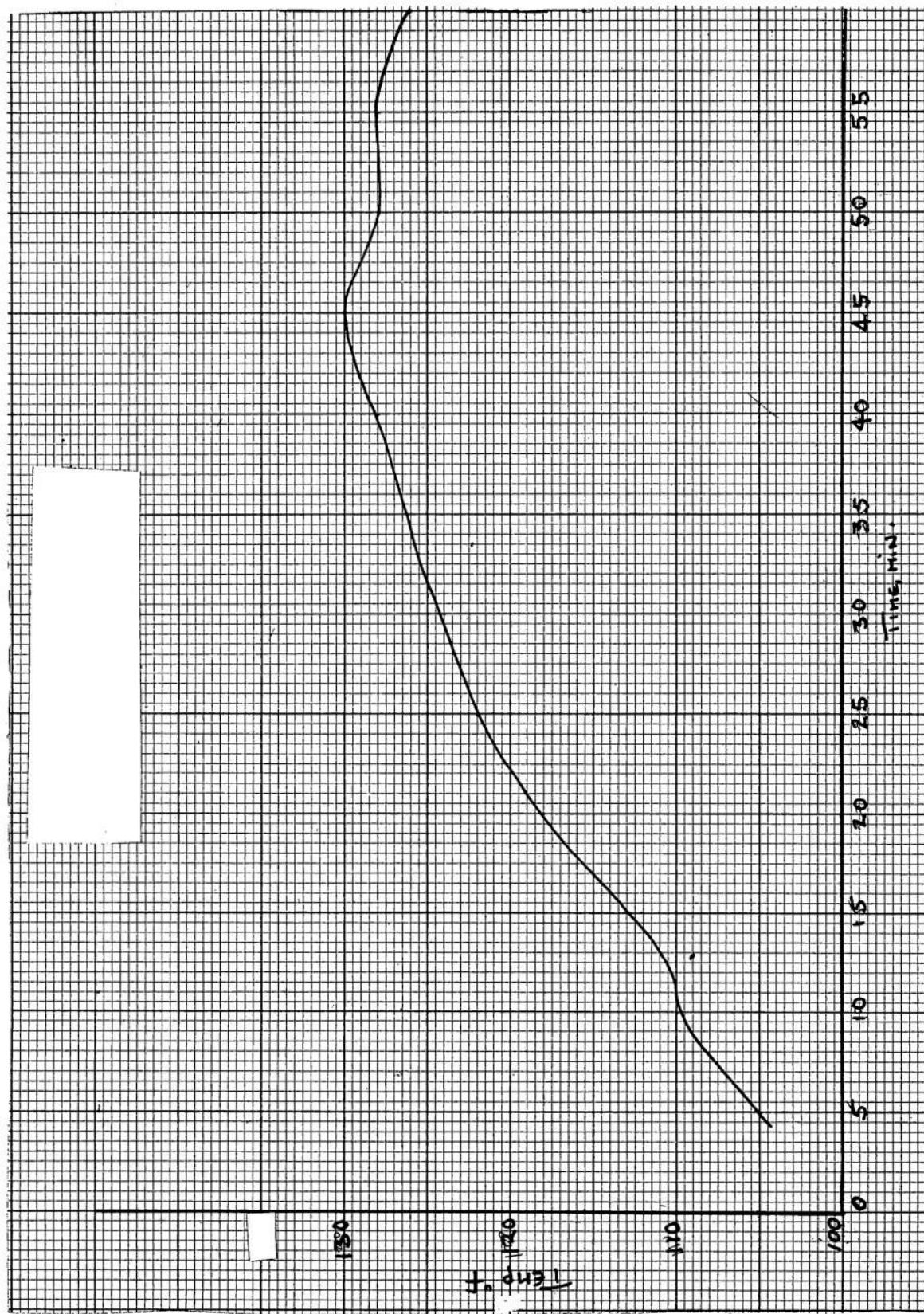


Figure 23. Exotherm curve of polyurethane before removal from mold—
room-temperature (26.7°C , 80°F) cured

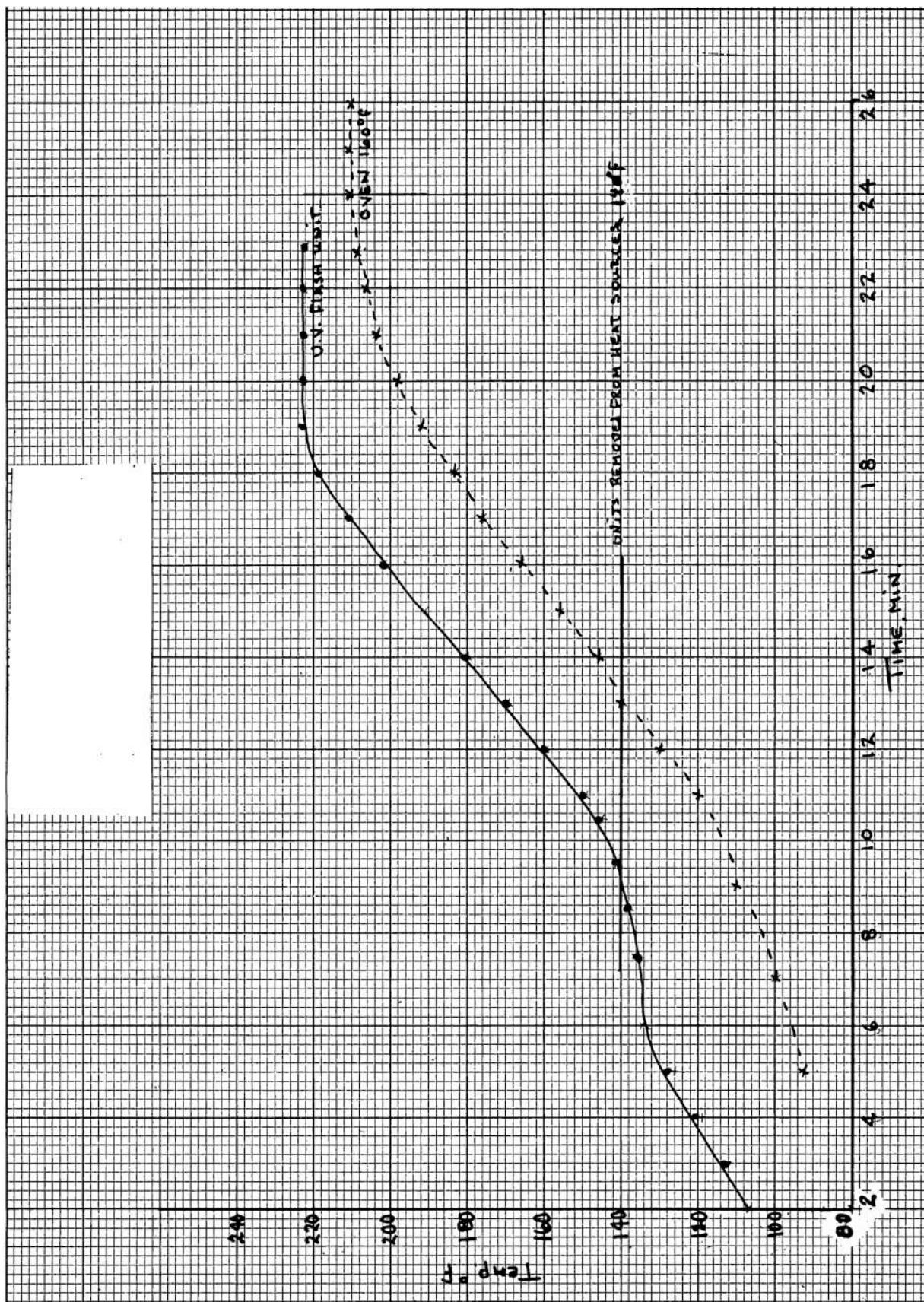


Figure 24. Exotherm curves of polyurethane encapsulated assemblies without battery cases and without circuit boards--ultraviolet- and oven-cured

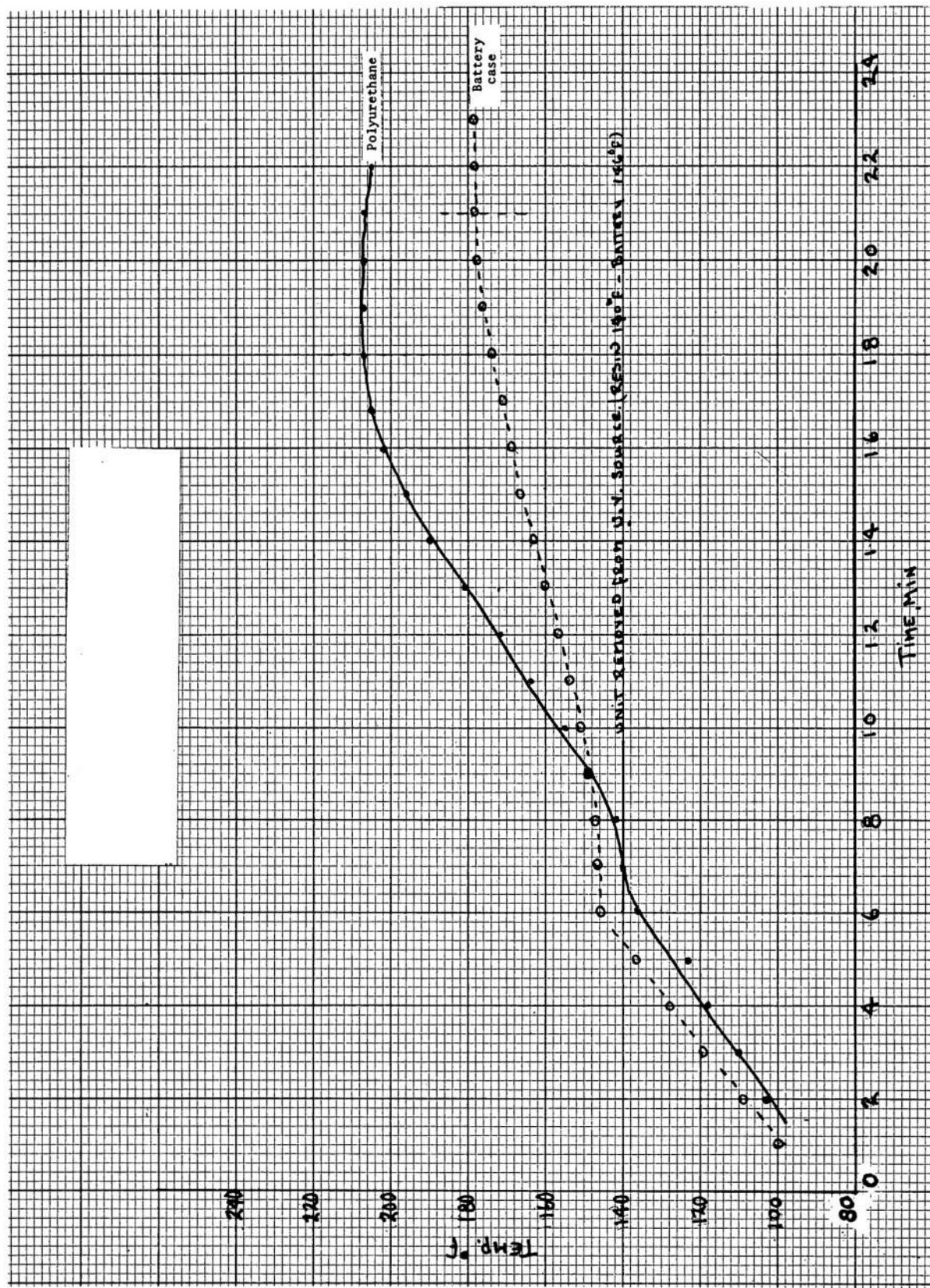


Figure 25. Exotherm curves of polyurethane encapsulated assemblies with battery cases and with circuit boards--ultraviolet-cured

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